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Tank 241-T-111 Characterization Report

B. C. Simpson

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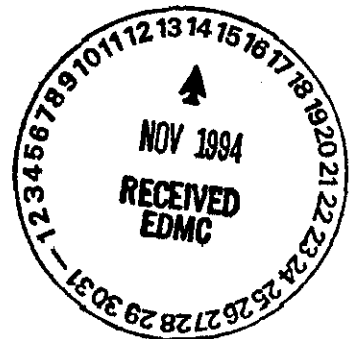
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Hanford Company

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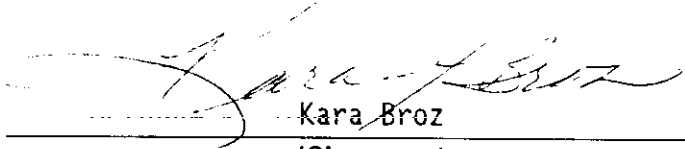
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EXECUTIVE SUMMARY

Single-shell tank 241-T-111 is a Hanford-Site High-Level Waste tank that was most recently sampled in late October and early November 1991. Analyses of materials obtained from tank 241-T-111 were conducted to support the Hanford Federal Facility Agreement and Consent Order¹ (Tri-Party Agreement) Milestone M-10-06 (now M-44-05). The waste in tank 241-T-111 is a complex material primarily made up of water and inorganic salts in a gel-like matrix. The insoluble solids are a mixture of phosphates, silicates, oxides, and hydroxides in combination with lanthanum, calcium, manganese, iron, bismuth, uranium, and chromium. The soluble analytes are primarily sodium, nitrate, sulfate, and fluoride (see Table ES-1).

Exotherms over -215 cal/dry gram were detected in the first two segments from each core. As a result of these analyses, tank 241-T-111 has been placed on the Organic Watch List². The source of these exotherms remains under investigation; however, under present tank conditions, there is no possibility that a rapid exothermic reaction will occur. Another finding from the characterization analyses was that the present method for determining or measuring total organic carbon was not effective for the organic materials in this waste matrix. Further investigation of the method failure for this waste is underway and other assay methods are being considered.

¹Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order*, 2 vols. as amended, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

²Wicks, J. H. 1994, *Tank T-111*, (Internal Memo 94-003 to D. A. Turner, March 3) Westinghouse Hanford Company, Richland, Washington.

Comparisons of the calculated bulk inventories for various analytes of concern show that tank 241-T-111 is within established operating safety requirements for heat-load, ferrocyanide, and plutonium inventory. From assessment of past and present surveillance data, tank 241-T-111 is considered an assumed leaking tank (see Table ES-2). Mitigation presently is taking place to remove the remaining free liquid in the tank in order to forestall any further leakage of tank contents to the environment. The free liquid is being transferred to tank 241-SY-102 as part of the overall single-shell tank stabilization effort³.

The results of the analyses have been compared to the dangerous waste codes in the "Washington Dangerous Waste Regulations" (WAC 173-303)⁴. This assessment was conducted by comparing tank analyses against dangerous waste characteristics ("D" waste codes) and against state waste codes. The comparison did not include checking tank analyses against "U," "P," "F," or "K" waste codes because application of these codes is dependent on the source of the waste and not on particular constituent concentrations. The results indicate that the waste in this tank is adequately described in the Dangerous Waste Permit Application for the Single-Shell Tank System; this permit is discussed in the Tank Characterization Reference Guide.⁵

³Jenkins, C. E. and D. B. Engleman, 1994, *Engineering Report: Managing the Assumed Leak from Single-Shell Tank 241-T-111*, WHC-SD-WM-ER-337, Westinghouse Hanford Company, Richland, Washington.

⁴WAC 173-303, "Dangerous Waste Regulations", *Washington Administrative Code*, as amended, Olympia, Washington.

⁵De Lorenzo, D. S., et al. 1994, *Tank Characterization Reference Guide*, WHC-SD-WM-TI-648, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Analysis of the process history of the tank provided valuable information about the likely physical and chemical condition of the waste. Direct comparisons with historical tank reviews underway at Los Alamos National Laboratory are not possible at this time because the Los Alamos National Laboratory effort has not progressed to encompass tank 241-T-111. However, estimates and comparisons using the available process knowledge⁶ have been made. Further comparisons with the Los Alamos National Laboratory database will be made when it becomes available in 1995. The available historical information, in combination with the analysis of the tank waste, supports the conclusion that a rapid exothermic reaction in tank 241-T-111 with the present tank conditions is not plausible because of the high moisture content of the waste and lack of any concentrated heat source. Therefore, the contents of tank 241-T-111 present no credible imminent threat to the workers at the Hanford Site, the public, or the environment. Because an exothermic reaction is not credible, the consequences of this accident scenario, as promulgated by the General Accounting Office, are not applicable.⁷ However, until the source and mechanism of the exotherm is further clarified, it was considered prudent to list the tank on the Organics Watch List, apply more rigorous access controls, and conduct further intrusive operations in tank 241-T-111 with greater care.

⁶Agnew, S. F., 1994, *Hanford Defined Wastes: Chemical and Radionuclide Compositions*, LA-UR-94-2657, Los Alamos National Laboratory, Los Alamos, New Mexico.

⁷Peach, J. D., 1990, *Consequences of Explosion of Hanford's Single-Shell Tank are Understated*, (Letter B-241479 to C. M. Synar, Chairman of Environment, Energy, and Natural Resources Subcommittee, Committee on Government Operations, House of Representatives), GAO/RCED-91-34, General Accounting Office, Washington, D.C.

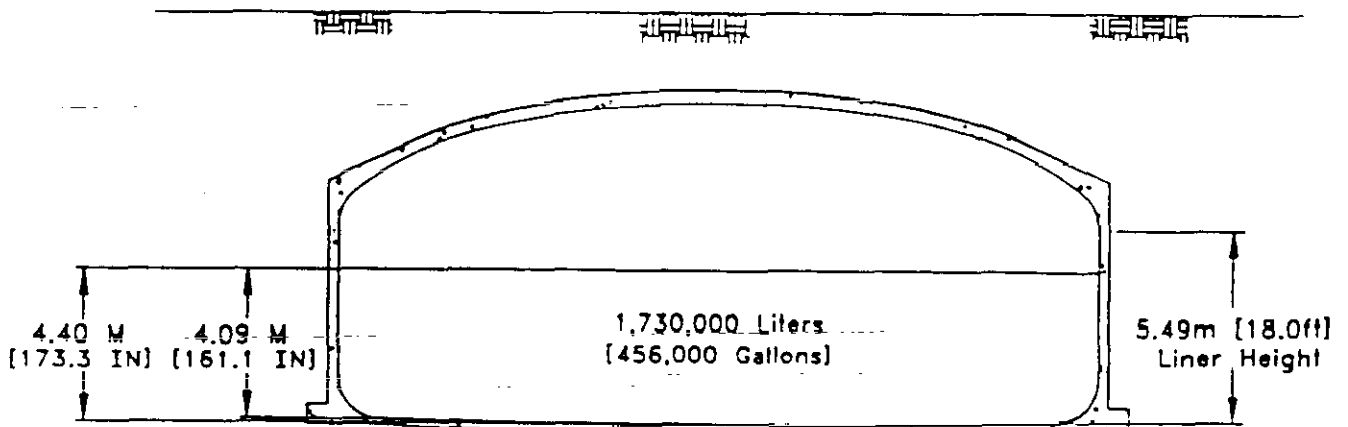
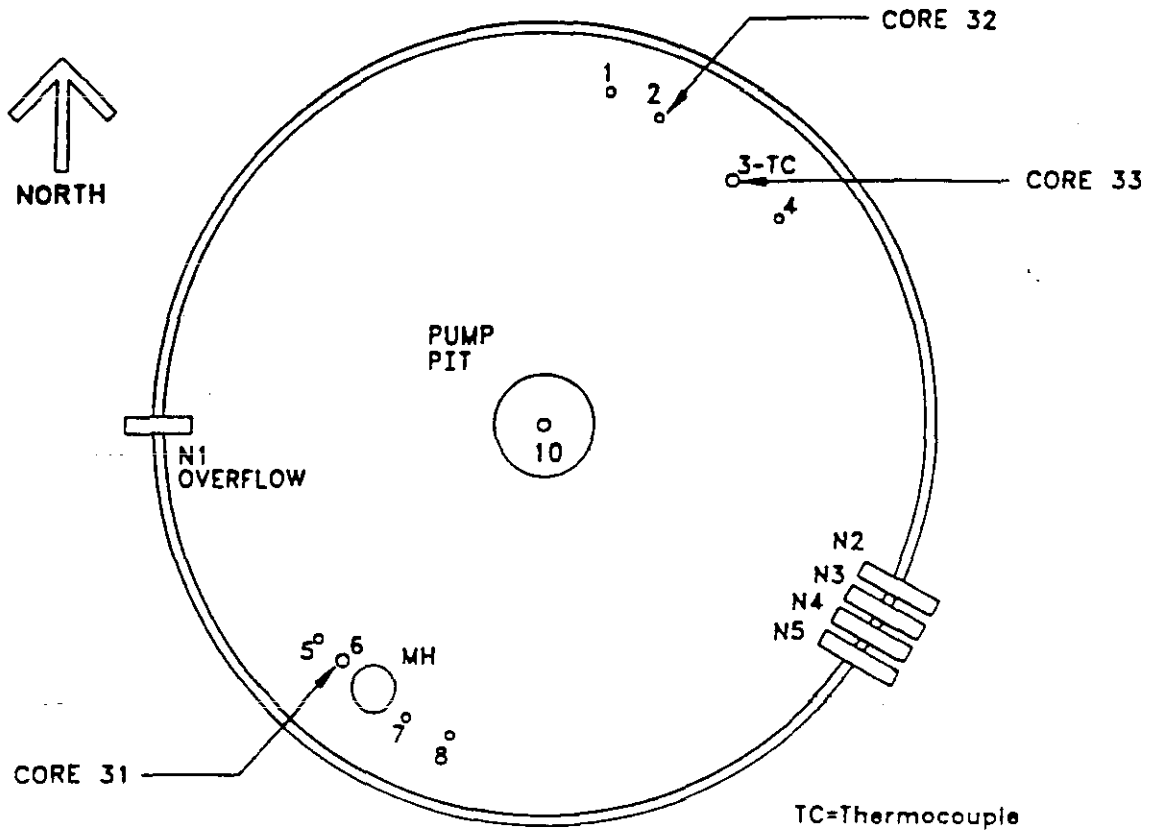
Table ES-1. Single-Shell Tank 241-T-111 Concentrations and Inventories for Critical List Analytes.

Physical Properties		
Density 1.16-1.28 g/mL	H ₂ O 76.0%	1,650,000 kg
Temperature 16 °C	Heat Load 81 w	(24 BTU/hr)
pH 11.65	Total Waste Mass	2,171,000 kg
Chemical Constituents	Average Concentration (wt%)	Bulk Inventory (kg)
Calcium (Ca)	0.242	5,260
Chromium (Cr)	0.198	4,290
Iron (Fe)	1.85	40,200
Manganese (Mn)	0.633	13,700
Sodium (Na)	3.70	80,300
Bismuth (Bi)	2.60	56,300
Lanthanum (La)	0.422	9,200
Silicon (Si)	0.567	12,300
Uranium (U)	0.355	7,700
Total Phosphate (PO ₄ ⁻³)	3.23	70,100
Sulfate (SO ₄ ⁻²)	0.355	7,700
Nitrate (NO ₃ ⁻)	4.13	89,700
Fluoride (F)	0.230	4,990
Total Organic Carbon (TOC)	0.312	6,770
Radionuclides	(μCi/g)	(Ci)
Total Plutonium	0.304	660
Am-241	0.0425	92.4
Sr-90	5.41	11,800
Cs-137	0.166	360

Table ES-2. Tank 241-T-111.

Tank Description	
Type:	Single-Shell
Constructed:	1944
In Service:	1945
Diameter:	75 ft (22.9 m)
Usable Depth:	17 ft (5.2 m)
Operating Capacity:	530,000 gal (2.01E+06 L)
Bottom Shape:	Dished
Hanford Coordinates:	43.347° North 75.737° West
Ventilation:	Passive
Tank Status: as of May 1994	
Contents:	Non-Complexed Waste
Total Waste:	456,000 gal (1.73E+06 L)
Supernate Volume:	0 gal (0 L)
Drainable Interstitial Liquid:	51,000 gal (193,000 L)
Manual Tape Surface Level (under riser):	-161.1 in. (408.9 cm)
Liquid Observation Well Level:	158.8 in. (403.4 cm)
Integrity Category:	Assumed Leaker
Watch List Status:	Organic

Figure ES-1.



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LIST OF ACRONYMS/ABBREVIATIONS

2C	Second-Cycle Decontamination Waste From The Bismuth Phosphate Process
224	Lanthanum Fluoride Waste
AA	Atomic Absorption
ANOVA	Analysis Of Variance
CI	Confidence Interval
DSC	Differential Scanning Calorimetry
GEA	Gamma Energy Analysis
IC	Ion Chromatography
ICP	Inductively Coupled Plasma - Atomic Emission Spectroscopy
PNL	Pacific Northwest Laboratory
RPD	Relative Percent Difference
SST	Single-Shell Tank
TGA	Thermogravimetric Analysis
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
Tri-Party Agreement	<i>Hanford Federal Facility Agreement And Consent Order</i>

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TANK CHARACTERIZATION REPORT: TANK 241-T-111

1.0 INTRODUCTION

In late October and early November of 1991, single-shell tank (SST) 241-T-111 was sampled and analyses were conducted on the materials obtained to complete *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) Milestone M-10-00 (Ecology et al. 1992) to sample and analyze two cores from each tank. Other objectives that these measurements and inventory estimates support are as follows.

- Obtain estimates of both the concentration and total quantity of key analytes relating to safety issues, such as organics and radionuclides.
- Provide input to risk assessment-based disposal decisions for the waste.
- Implement physical property measurements, such as rheology, bulk density, and particle size. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and vitrification systems.

1.1 PURPOSE

The purpose of the tank characterization report is to describe and characterize the waste in SST 241-T-111, based on information from various sources. This report summarizes the available information regarding the waste in tank 241-T-111, and arranges this information in a format useful to support management and technical decisions concerning this waste tank.

1.2 SCOPE

This report presents a broad background of information that was available before core sampling, which initially guided the development of the sampling and analysis program. This material includes process stream data, historical information about any previous characterization testing, transfer records, and observations from in-tank photographs. The results of tank 241-T-111 core-sample analyses are summarized and presented, along with a statistical interpretation of the data. The information obtained from historical sources will be compared and correlated with the actual waste measurements in this report. As characterization efforts proceed and additional information becomes available, this document will be revised periodically to reflect the new data set.

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2.0 HISTORICAL TANK INFORMATION AND EVALUATION

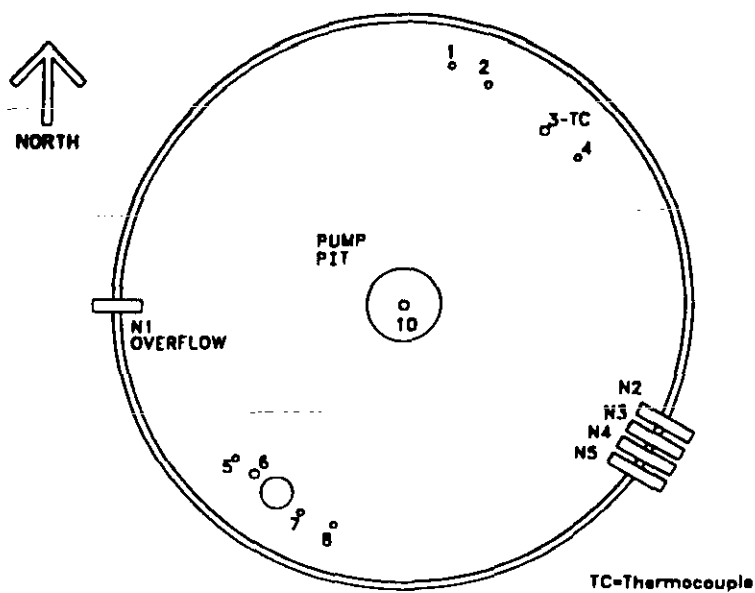
2.1 BACKGROUND

Radioactive wastes from defense operations have accumulated at the Hanford Site in underground waste tanks since the late 1940s. The original tank farms (B, C, T, and U) were built from 1943 to 1944. Tank 241-T-111 was placed into service in 1945. Groups of waste tanks that were physically located together and built at the same time are called tank farms. Each original tank has a diameter of 22.9 m (75 ft), an operating depth of 5.2 m (17 ft), and a nominal capacity of 2 million liters (530,000 gal). The basic design of a typical SST is shown in Figure 2-1. The tanks were constructed of reinforced concrete with a mild steel liner covering their bottoms and sides. The carbon steel liners were designed to receive and contain neutralized, mildly alkaline wastes. The tops of the tanks are concrete domes. Tanks such as 241-T-111 were all covered by at least 1.8 m (6 ft) of soil for shielding purposes (Anderson 1990). The tanks in the tank farms were connected in groups of three or four and overflowed from one to another in a configuration known as a cascade. Tank 241-T-111 is the middle tank in a cascade that includes 241-T-110 and 241-T-112. Cascades served several functions in Hanford-Site waste management operations. By cascading tanks, fewer connections needed to be made during waste disposal. Consequently, all three tanks were usable without having to connect the active waste transfer line directly to each individual tank. This handling method reduced the likelihood for personnel exposure to the waste and diminished the chances for a loss of tank integrity because of overfilling. Another benefit of the cascades was clarification of the wastes. When used in this manner, most of the solids in the waste slurries routed to the tanks settled in the first tank (241-T-110), and the clarified liquids cascaded on to the other tanks in the series (241-T-111 and 241-T-112). Supernate from the final tank in a cascade series was sometimes routed to a disposal trench. Since most radionuclides are insoluble in aqueous alkaline media, clarification reduced the potential amount of radiological contamination to the environment. However, historical sources report that cascade lines routinely clogged (Anderson 1990). When clogging occurred, very little could be done to resolve the problem, other than re-routing the effluent stream directly to the disposal tank. Cascading was a common practice in the early-process history of the tanks, but became less frequent as time passed, virtually ceasing by the late 1950s.

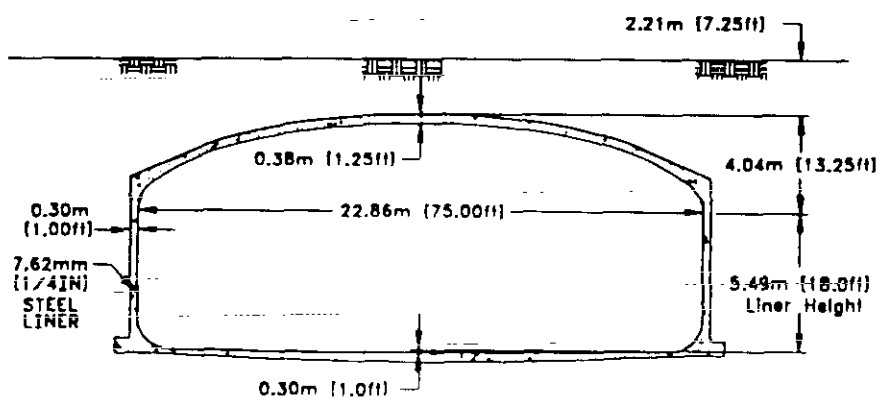
2.2 TANK 241-T-111 HISTORY

The first type of waste that tank 241-T-111 received and stored was second-cycle decontamination waste from the bismuth phosphate process (2C) (1945 to 1952). The tank was filled by a cascade of 2C waste in the fourth quarter of 1945. The supernatant in the tank was disposed to a crib in the third and fourth quarters of 1947. The tank was then re-filled with 2C waste in the second quarter of 1948. After the cascade was filled again in late 1948, tank 241-T-111 remained in active service. From 1953 to 1955, tank 241-T-111 was

Figure 2-1. Typical Single-Shell Tank Diagram.



TANK RISER LOCATION



Note: Tank 241-T-111 has a dished bottom.

used to cascade ^{235}U and lanthanum fluoride waste (224) from the LaF_3 finishing process in T Plant to a crib. In addition, Anderson (1990) reports that some 5-6 cell drainage out of B Plant was routed to the tank along with 224 waste in 1952.

After the end of the T-Plant cascade in 1955, the reported total waste volume remained relatively unchanged [between 1.98 and 2.12 million L (524,000 and 560,000 gal)] for the remainder of the tank's active service life. There was a residual heel of at least 1.85 million L (488,000 gal) left from previous waste management operations. Tank 241-T-111 remained in 2C service through the third quarter of 1956 (T Plant ran BiPO_4 until August 1956). Anderson (1990) notes that in 1952 the tank was also receiving 224 waste from the plutonium purification/concentration processes performed in the 224-T building.

T Plant initially was built as a bismuth phosphate processing plant, however that purpose was changed when it became an equipment decontamination facility. The tank would have also received miscellaneous decontamination chemicals from T-Plant decontamination operations performed in the 1960s. The records are not clear whether tank 241-T-111 always received waste as overflow from tank 241-T-110, or if waste was later routed directly to it. There is anecdotal evidence that the cascade overflow line plugged early in the service life of tank 241-T-110, and that a direct discharge line from T Plant was used to dispose of wastes to tank 241-T-111, however no reference confirming this fact can be found. Between 1964 and 1974, the reported solids volume fluctuated widely between 1.93 and 0.88 million L (510,000 and 233,000 gal). A highly anomalous reading of 150,000 L (40,000 gal) is not considered credible and is believed to be the result of a transcription error. In that time period, the total volume reported rose slightly, then decreased from 2.06 million L to 1.85 million L (541,000 to 488,000 gal) (Anderson 1990). In the second quarter of 1974, there were two small transfers out of Tank 241-T-111: one of 106,000 L (28,000 gal) to tank 241-S-110 and one of 53,000 L (14,000 gal) to tank 241-T-109.

In the third quarter of the same year, dry wells 50-11-05 and 50-11-08 were drilled and in the fourth quarter of 1974, tank 241-T-111 was removed from active service. In the first and second quarters of 1976, two minor transfers of 30,300 L and 19,000 L (8,000 and 5,000 gal), respectively, were made out of tank 241-T-111. Saltwell pumping commenced in the third quarter of 1976 as part of the tank stabilization effort, and no further waste receipts were made. In 1979, the integrity of the tank was questioned and dry well 50-11-11 was drilled in the third quarter. As a result of an observed level drop, tank 241-T-111 was declared an assumed leaker in 1984 (Hanlon 1994). Figure 2-2 illustrates the fill and transfer history of 241-T-111.

The wide fluctuation in the early reported solids level makes it difficult to derive any firm conclusions regarding the stratification in the tank on a strictly historical basis. Overall sludge volume in the tank may have decreased somewhat between 1956 and 1974 with further settling and compaction from the weight of overlying solids. The amount of sludge added since the end of the T-Plant cascade activity probably is negligible, because the transfer history of the tank was so limited. Floating suction pumps do not transfer solids

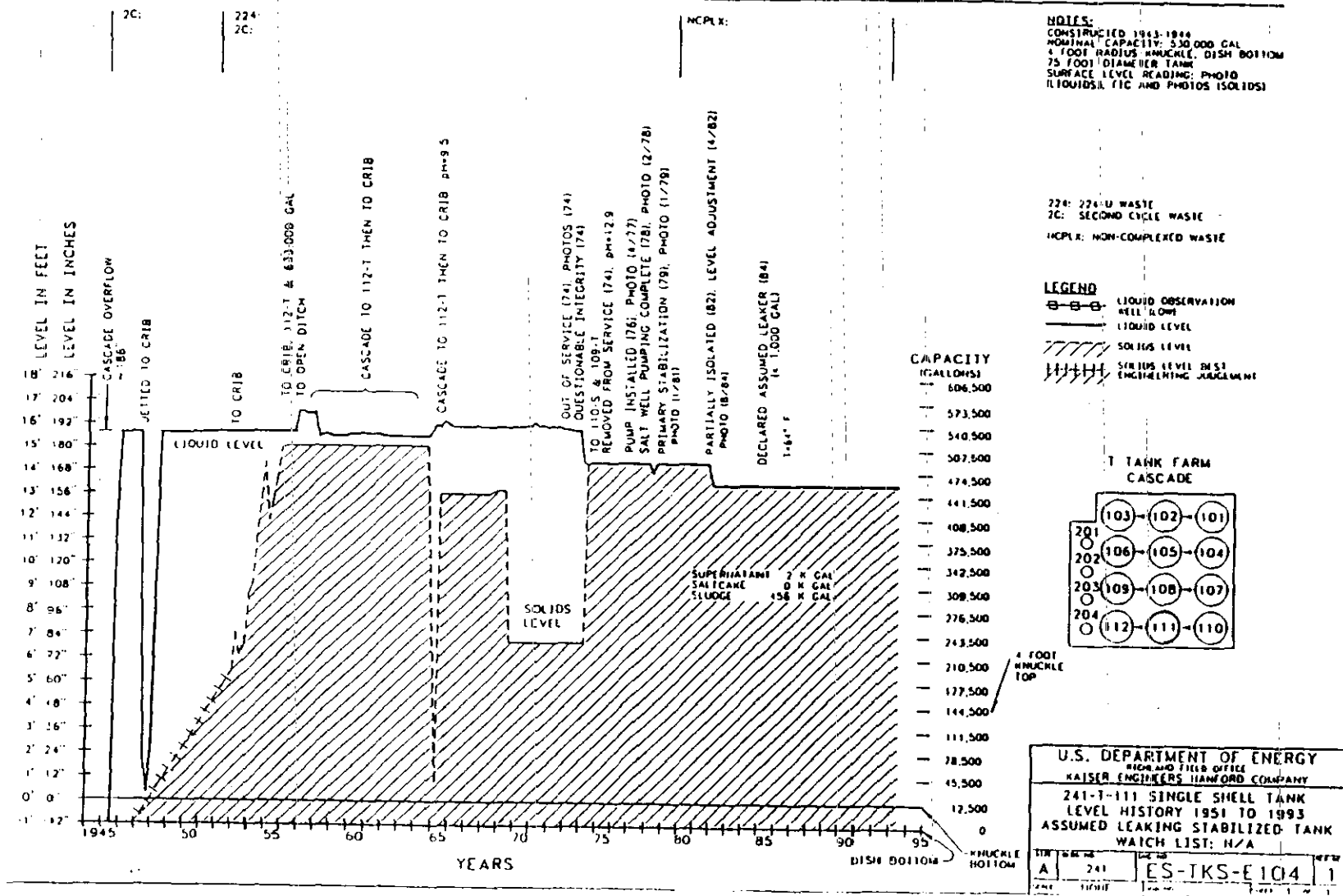


Figure 2-2. Fill and Transfer History of 241-T-111.

readily, and the movement of more than 950,000 L (250,000 gal) of solids in a quarter seems unlikely. However, the reported solids measurement in the fourth quarter of 1956 [1.93 million L (510,000 gal)] and the reported solids measurement at the end of the tank's active service life in 1980 [1.85 million L (488,000 gal)] appears to be reasonable, a 4.5 percent difference, given the time and compaction processes ongoing in the tank, and the inherent uncertainties associated with early solids measurements in the tank farms.

2.3 PROCESS KNOWLEDGE

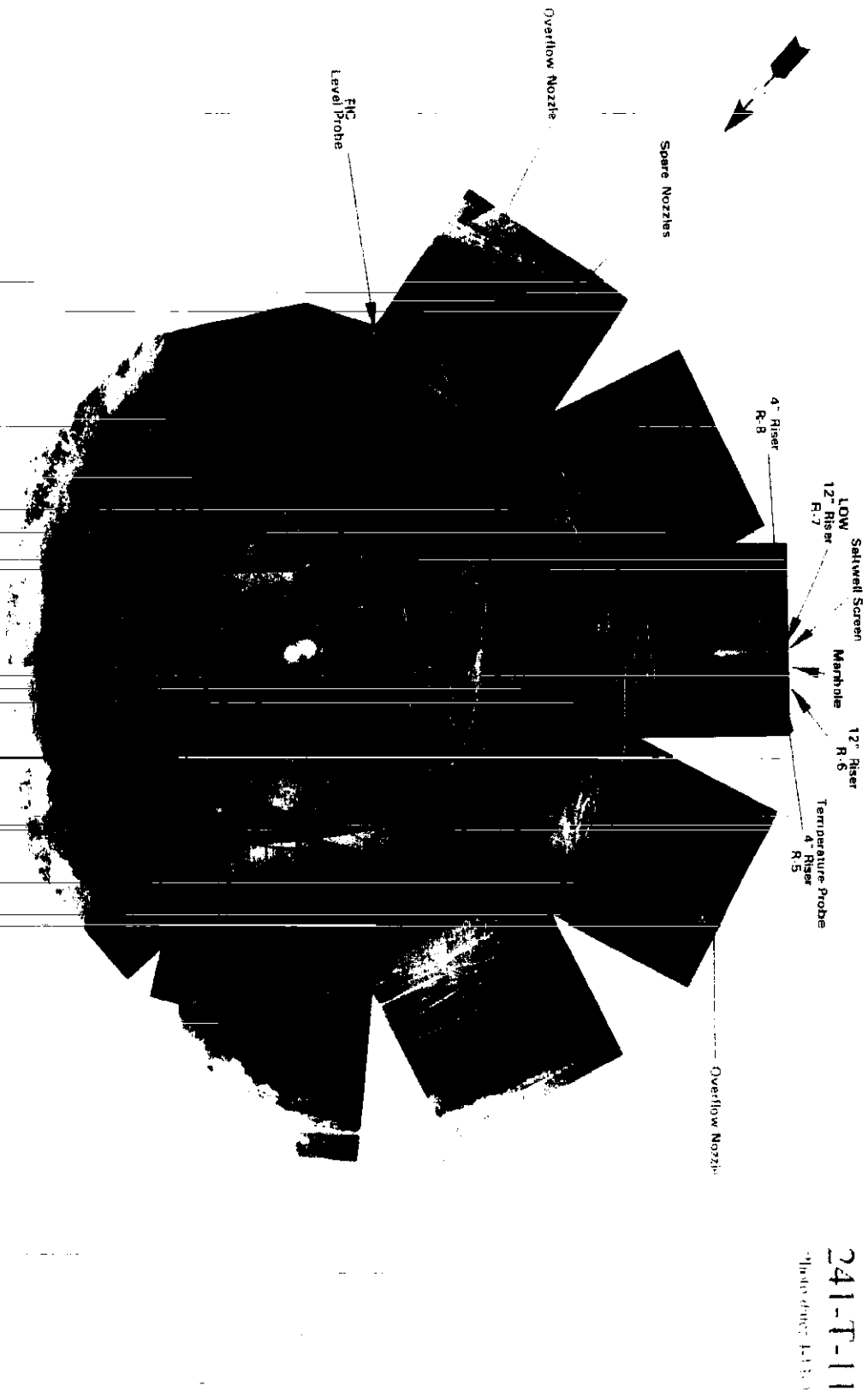
Process knowledge obtained from historical records can be used to predict the major constituents and some general physical properties of the waste matrix in the tanks. Tank 241-T-111 is expected to contain relatively soft sludge, which can be push-mode sampled. During its operating history, tank 241-T-111 was never subject to any of the various waste volume reduction or in-tank solidification processes. Consequently, there was no formation of hard salt cake on top of the sludge, as there was in the BY or TX Tank Farms (Anderson 1990). This expectation was supported by inspection of in-tank photographs that indicated a moist and pliant waste surface (see Figures 2-3 and 2-4). The effluents that were added to the tank during waste management operations were slurries consisting primarily of water (Schneider 1951). There was no mixing equipment in tank 241-T-111 to blend the layers of settled solids together and there were distinct differences in the composition of wastes directed to it over its operating life. Agnew (1994) predicts that 2C and 224 wastes are not identical in makeup, and analytically observable variations in composition are expected. Because of the lack of agitation or mixing in the tank, observable evidence of layering is expected in some of the segment-level analytical results.

Previous analytical data on tank 241-T-111 is scant. The results of some liquid samples taken in 1974 are provided in Appendix C. Because the tank has been pumped several times since those samples were taken, they are not considered representative of the present waste matrix. The lower solids in tank 241-B-110 should be similar to the lower solids in tank 241-T-111 (Borsheim 1994). Both tanks were filled at least twice with 2C. Tank 241-B-110 was the first tank in the B-Plant 2C cascade while tank 241-T-111 was the second tank in the T-Plant 2C cascade. Further studies and comparisons of tank compositions will be forthcoming, pending additional analytical information on the various waste types and matrices that exist in the tank farms. The solids from the neutralized solution would have settled out in the tanks while the supernatant was disposed to cribs. The upper solids of tank 241-T-111 should have some similarity to the B and T 200-series tanks, because they all received 224 waste.

The estimated composition of neutralized 2C and 224 waste streams (i.e. unsettled) are given in Table 2-1 as determined from Schneider (1951). The Schneider (1951) process stream compositions are for the unsettled wastes being transferred from the separations plants. The

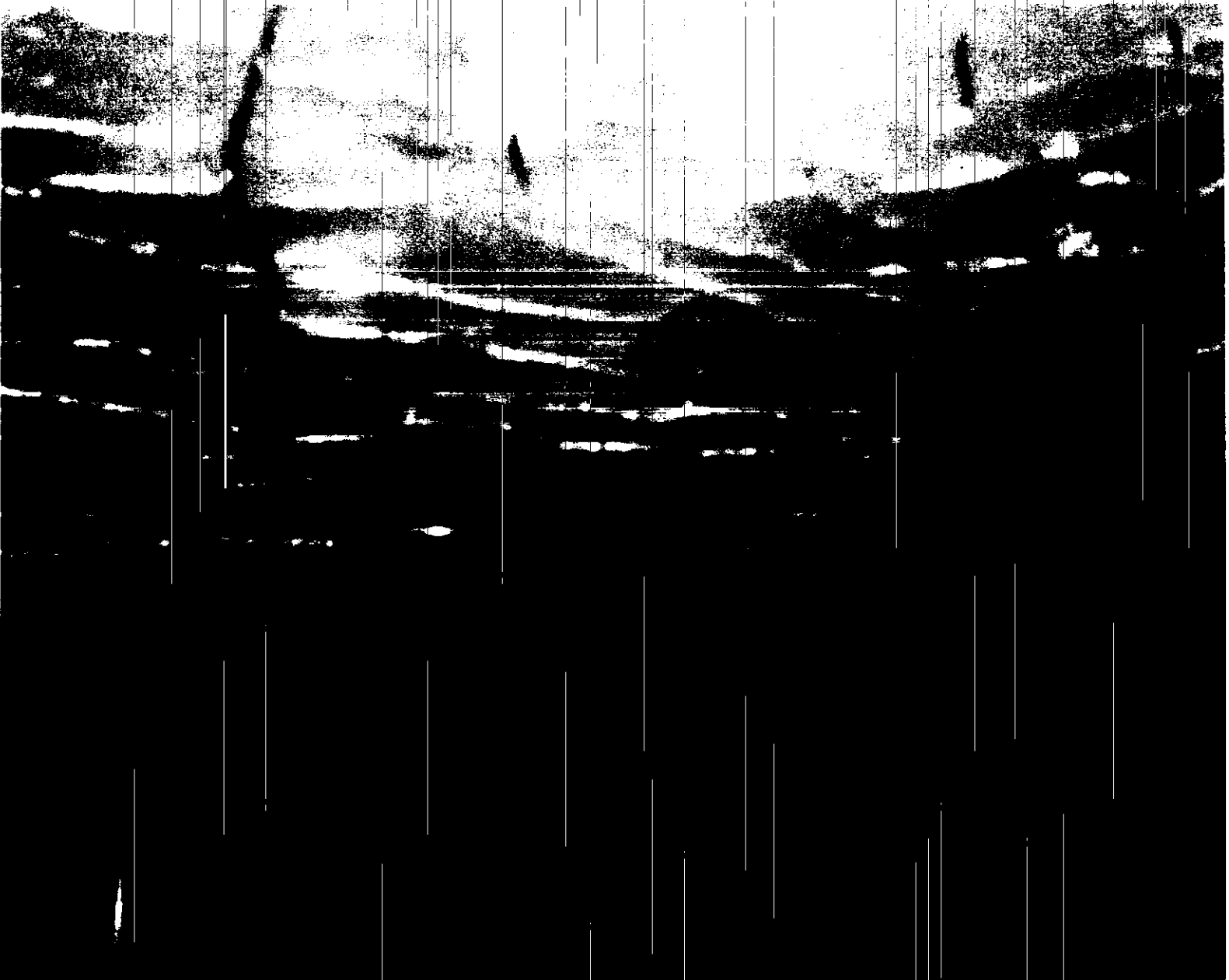
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Figure 2-3. 241-T-111 Surveillance Photo - Collage.



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Figure 2-4. 241-T-111 Surveillance Photo - Close up.



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estimates using Agnew (1994) provide insight to the analyte concentrations of the settled sludge. How the waste settled, the solids content of the settled waste, and how it affected the waste inventory of the tank will be discussed further in Section 6.

Table 2-1. Typical Waste Stream Compositions and Expected Sludge Compositions for Selected Analytes (Wet Basis).

Analyte	Unsettled 2C BiPO ₄ waste (Schneider 1951)		Settled 2C BiPO ₄ waste, (Agnew 1994) average 1944-1951 and 1951-1956 compositions		Unsettled BiPO ₄ -LaF ₃ 224 process waste (Schneider 1951)		Settled BiPO ₄ -LaF ₃ 224 process waste (Agnew 1994)	
	Wt %	(μg/g)	Wt %	(μg/g)	Wt %	(μg/g)	Wt %	(μg/g)
Bi	0.12	1,200	2.55	25,500	0.11	1,100	2.07	20,690
Cr	0.006	60	0.0033	33	0.016	160	0.0035	35
Na*	3.5	35,000	6.46	64,600	3.39	33,900	8.50	85,000
NH ₄ ***	0.16	1,600	-	-	0.011	110	-	-
Fe	0.17	1,700	1.79	17,900	-	-	0	0
Mn	-	-	0	0	0.031	310	0.019	190
K	-	-	0	0	0.79	7,900	0.779	7,790
La	-	-	0	0	0.05	500	2.67	26,700
Anions								
PO ₄ ³⁻	2.2	22,000	5.63	56,300	0.28	2,800	1.25	12,500
SO ₄ ²⁻	0.34	3,400	0.276	2,760	0.032	320	0	0
NO ₃	5.8	58,000	4.28	42,800	3.91	39,100	6.16	61,600
F	-	-	0.494	4,940	0.52	5,200	4.66	46,600
SiF ₆ ²⁻	0.35	3,500	-	-	-	-	-	-
C ₂ O ₄ ²⁻	-	-	-	0	0.12	1,200	1.52	15,200
H ₂ O	87.3	873,000	76.0	760,000	90.75	907,500	69.0	689,600

*Analytes listed in italics are mostly soluble.

**NH₄⁺ probably has dissipated over time and is believed to be no longer present.

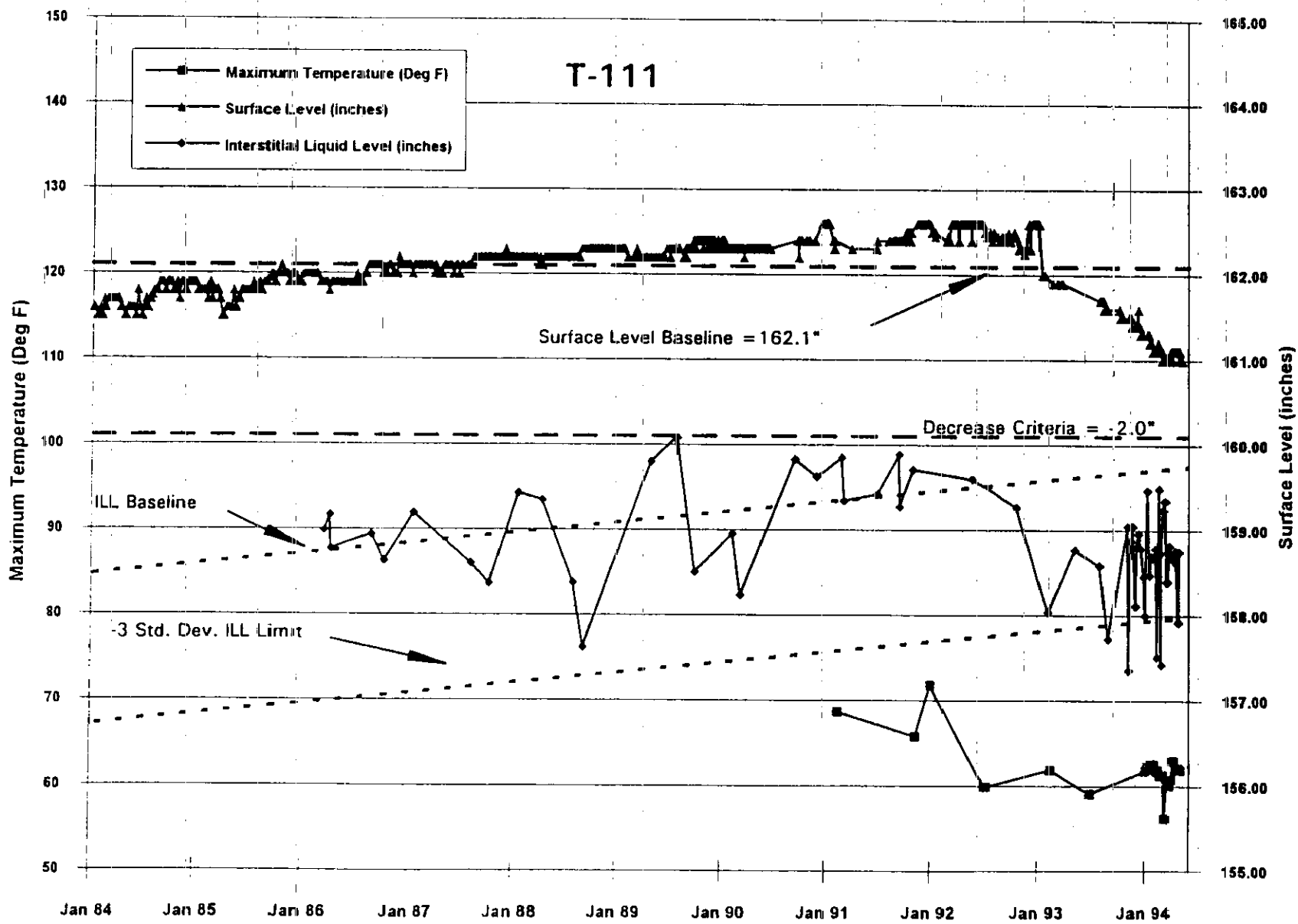
2.4 SURVEILLANCE DATA

The most recent waste inventory measurement for tank 241-T-111 reports approximately 1.73 million L (456,000 gal) of solid waste with an estimated 193,000 L (51,000 gal) of drainable liquids (Hanlon 1994). The most recent surveillance data shows a discernable downward trend over the past 18 months, nearly a 3.8 cm drop in surface level, after a very long, gradual, but minor upward trend over the last ten years (see Figure 2-5). This recent behavior possibly indicates further settling, or leaking has taken place. These figures translate to a waste depth of 408.9 cm (161.1 in.) underneath the riser and 440.2 cm (173.3 in.) at the tank centerline. The tank has not been fully interim stabilized yet, and is an assumed leaker. The recent waste temperature in tank 241-T-111, taken from a thermocouple tree is approximately 16 °C (60.5 °F) (Rios 1994), and the estimated heat load in the tank is less than 2.93 kW (10,000 Btu/hr).

2.5 TANK STATUS

Tank 241-T-111 was a non-watch-list SST, with no historical indication of any potential safety issue. However, during the review of the energetics data that was done in support of characterization, exotherms in excess of -125 cal/dry gram of waste were noticed in the top 3 segments of core 31 and the top 2 segments of core 33. Additional follow-up work has been done confirming the initial observation and as a result, 241-T-111 has been added to the Organic Tank Watch List (Wicks 1994). The exotherms were not predicted from the process history of the tank and the known characteristics of the 2C and 224 waste streams disposed there.

Figure 2-5. 241-T-111 Surveillance Data.



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3.0 TANK SAMPLING OVERVIEW

3.1 DESCRIPTION OF SAMPLING EVENT

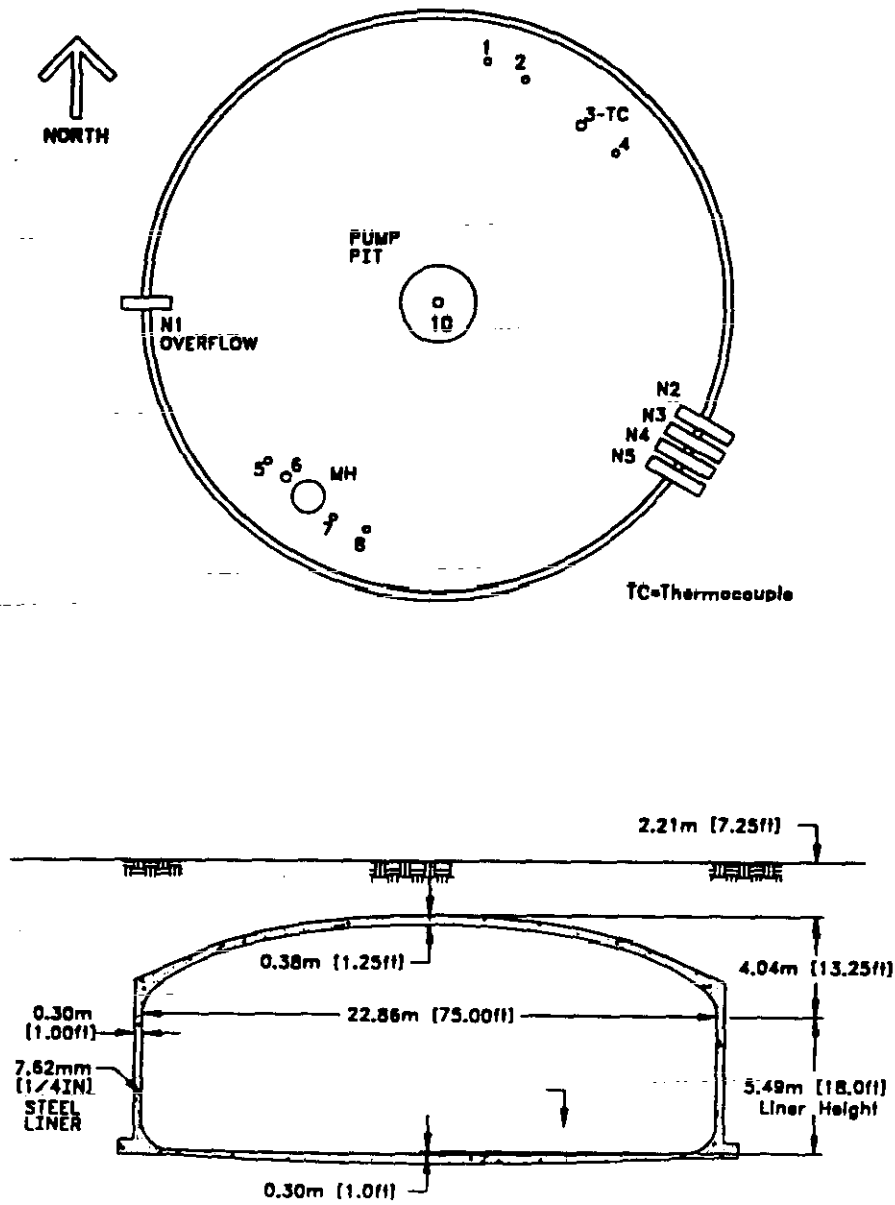
Tank 241-T-111 was push-mode core sampled through three risers during a period from October 22, 1991 to November 7, 1991. The core samples were obtained using a specially designed core sampling truck. Access to the interior of the tank is provided by various tank risers. Risers are pipes of various diameters leading into the tank dome from the ground. The riser configuration for tank 241-T-111 is given in Figure 3-1. A review of the tank farm operating records and a field inspection of the tank risers determine which risers can be used in the sampling operation. During sampling, a riser is opened and the truck is positioned over the riser. The sampler is lowered into the tank through the drill string and pushed into the waste. Further information regarding the core sampling operation can be found in *Tank Farm Operating Procedure* (Ross 1993). Nine segments were expected from each core sample. Each segment is approximately 48 cm (19 in) long. Core 31 was obtained from riser 6 on October 22, 1991. Core 32 was obtained from riser 2 from October 24, 1991 to October 25, 1991. Core 33 was obtained from riser 3 on November 5, 1991 to November 7, 1991.

The sampler is constructed of stainless steel and is 48 cm (19 in) long, with a 2.2-cm (7/8-in) inside diameter, and has a volume of 187 mL (0.05 gal). A hydrostatic fluid of normal paraffin hydrocarbons, similar to kerosene, was used in establishing a head balance while taking these cores. Objections involving sample degradation and contamination have been raised regarding the use of this fluid, and the practice has since been discontinued. However, for cores 31 and 33, nearly full recovery was achieved in every case. There were little or no drainable liquids observed in the sample liners or in the samplers upon extrusion of the samples, and although hydraulic permeability measurements were not taken as part of the characterization effort, the waste did not appear porous. Thus, sample contamination from the hydrostatic fluid is not deemed to be a significant issue with the analysis of the sample or the interpretation of the results.

The casks were transported to the 222-S Laboratory for characterization analysis. This facility is operated by Westinghouse Hanford Company in the 200 West Area of the Hanford Site. Further physical and radiochemical characterization was performed at the 325 Laboratory. Specific analyses aimed at identifying and resolving the unusual energetics observed (Bean 1994) are also being done at 325 Laboratory. That facility is operated by Battelle Pacific Northwest Laboratory (PNL), and is located in the 300 Area of the Hanford Site.

Before the most recent stabilization effort, a series of liquid grab samples were taken and analyzed to ensure waste compatibility with the tank receiving the 241-T-111 liquid waste. These grab samples were acquired using a sampling method known as "bottle-on-a-string," in which a weighted bottle with a shuttered mouth is lowered into the liquid waste. The bottle's

Figure 3-1. Tank 241-T-111 Riser Configuration.



mouth is opened, it fills with liquid, the mouth is closed, and the sample is retrieved and transported to the 222-S Laboratory. Winters et al. (1990a and 1990b) has a more detailed description of this sample method.

3.2 CHAIN OF CUSTODY

A chain-of-custody record was kept during the sampling event for each segment that was sampled. The chain-of-custody form is a one-page record that is used to ensure that (1) the sample is safely and properly transported from the field to the laboratory, and (2) the correct personnel are involved in the sampling operation and transportation of the sample to the laboratory.

One of the additional functions of the chain-of-custody record is to provide radiation survey data. This is a record of the radiation dose that is emitted from the shipping cask. The dose rates in mrem/hour are measured from the top, sides, and bottom of the cask. These values are recorded on the chain-of-custody record and represent the radiation being emitted directly from the sample. The last item recorded under the radiation survey data is the smearable contamination. Smearable contamination represents the radiation from waste material that is not sealed within the shipping cask; values greater than 100 mrem/hour are considered unsafe. Measurements are made both in the field and in the laboratory. No smearable contamination was found with these samples.

The chain-of-custody has several other important functions: (1) to provide a modest description of the cask, sampler, and the expected contents of the sampler (shipment, sample, and cask serial numbers for the specific sampling event); (2) to provide summary information about the analytical suite that the sample will undergo or reference the salient documentation; (3) to provide traceability for the sample during transport; and (4) to ensure sample integrity on arrival at the laboratory. This information is provided to ensure that each sample can be uniquely identified. A summary of the most pertinent data contained in the chain-of-custody forms for the tank 241-T-111 samples is presented in Tables 3-1 and 3-2.

Copies of the chain-of-custody forms are available in the full data package and through Hanford-Site Central Files. From inspection of the chain-of-custody records, there appear to be irregularities in the sampling and transport of tank 241-T-111 samples. Valve failures were observed in individual segments in all three core samples, and core 32 was considered to be completely compromised and non-representative. Each segment was almost entirely aqueous, containing at most a small amount of suspended solids. Liquid was also found in some of the liners surrounding the samplers from core 32, and was assumed to be leakage from the sampler. These irregularities merit a sampling concern, sample integrity concern, and potential safety concern (i.e., sample containment was compromised). However, the double-containment strategy employed in the handling of the samples was successful in preventing any excessive radiological exposure to personnel and no material escaped confinement. Further investigation and refinement of the sampling process, procedures, and sampler design is in progress.

Table 3-1. Core 31--Chain of Custody Summary.

Sample	Core 31	
Place Taken	241-T-111 Riser 6	
Date Taken	10/22/91	
Date Released	10/23/91	
Time Released	9:35 P.M.	
Sender	D. C. Hartley	
Receiver	V. Johansen	
Place Received	222-S Laboratory	
Time Received	10:00 P.M.	
Sample Number	Smearable Contamination	Dose Rate Through the Drill String
91-090 (Segment 1)	< DL alpha < DL beta-gamma	4.5 mR/hr
91-091 (Segment 2)	< DL alpha < DL beta-gamma	2.4 mR/hr
91-092 (Segment 3)	< DL alpha < DL beta-gamma	2.5 mR/hr
91-093 (Segment 4)	< DL alpha < DL beta-gamma	2.0 mR/hr
91-094 (Segment 5)	< DL alpha < DL beta-gamma	1.5 mR/hr
91-095 (Segment 6)	< DL alpha < DL beta-gamma	1.5 mR/hr
91-096 (Segment 7)	< DL alpha < DL beta-gamma	0.5 mR/hr
91-097 (Segment 8)	< DL alpha < DL beta-gamma	1.5 mR/hr
91-098 (Segment 9)	< DL alpha < DL beta-gamma	0.3 mR/hr

< DL = below detection limit

Table 3-2. Core 33--Chain of Custody Summary

Sample	Core 33		
Place Taken	241-T-111 Riser 3		
Dates Taken	11/5/91 to 11/6/91		
Dates Released	11/6/91 to 11/7/91		
Sender	D. C. Hartley		
Receiver	V. Johansen		
Place Received	222-S Laboratory		
Times Received	See Below		
Sample Number/ Date Sampled	Date Released/ Time Released/ Time Received	Smearable Contamination	Dose Rate Through the Drill String
91-108 (Segment 1) 11/5/91	11/6/91 10:10 10:55	< DL alpha < DL beta-gamma	3 mR/hr
91-109 (Segment 2) 11/5/91	11/6/91 10:10 10:55	< DL alpha < DL beta-gamma	2.5 mR/hr
91-110 (Segment 3) 11/5/91	11/6/91 10:10 10:55	< DL alpha < DL beta-gamma	10 mR/hr
91-111 (Segment 4) 11/5/91	11/6/91 14:10 14:30	< DL alpha < DL beta-gamma	5 mR/hr
91-112 (Segment 5) 11/6/91	11/6/91 14:10 14:30	< DL alpha < DL beta-gamma	< 0.5 mR/hr
91-113 (Segment 6) 11/6/91	11/6/91 14:10 14:30	< DL alpha < DL beta-gamma	2 mR/hr
91-114 (Segment 7) 11/6/91	11/7/91 10:10 10:35	< DL alpha < DL beta-gamma	1.5 mR/hr
91-115 (Segment 8) 11/6/91	11/7/91 10:10 10:35	< DL alpha < DL beta-gamma	1 mR/hr
91-116 (Segment 9) 11/6/91	11/7/91 10:10 10:35	< DL alpha < DL beta-gamma	1 mR/hr

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4.0 SAMPLE HANDLING AND ANALYTICAL SCHEME

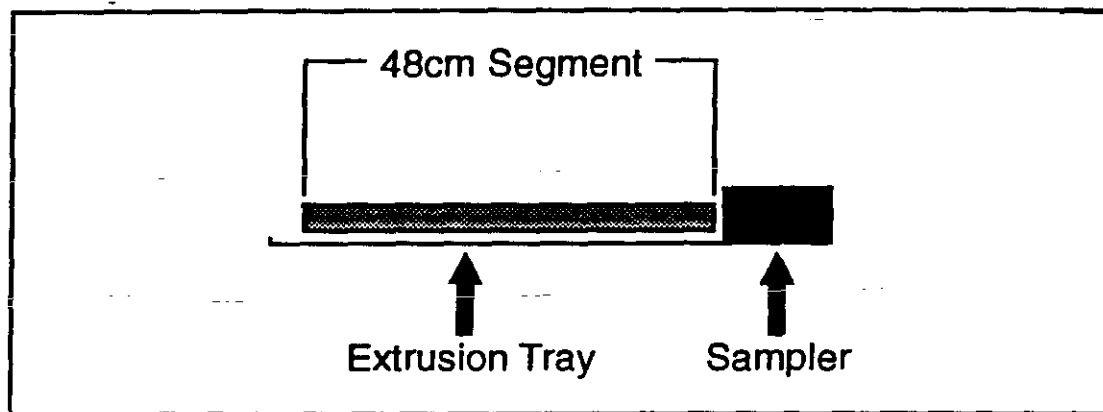
The primary objective for these waste analyses was to meet requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1992, Ecology et al. 1994). Two composites from each core were built and analyzed in accordance with the complete baseline case core composite scenario detailed in WHC-EP-0210 (Winters et al. 1990a, Winters et al. 1990b) and as amended by Hill et al. (1991). However, there are other concerns and interests for this data. First, this information will help to evaluate whether constituent concentrations are within safe operating limits by determining whether they are flammable or explosive. Second, analyte concentrations of interest to the various Hanford-Site technical programs can be determined.

Because tank 241-T-111 initially was identified as a non-watch-list tank, extensive analytical measurements were not specified to resolve any previously identified safety concerns associated with this tank. The analysis horizon for characterization was determined to be on the core composite level with selected analyses being performed on a segment-level basis. However, after the discovery of substantial exotherms in the top 100 cm (40 in) of the waste, additional testing on a segment level basis was done on those samples exhibiting reactive behavior. This additional testing, with an emphasis on providing resolution to the safety issues raised by the presence of the exotherms in the waste is still underway at the time of the writing of this report. This report will be updated to reflect the new data or root causes of the energetics when that information becomes available.

4.1 SAMPLE BREAKDOWN PROCEDURE

In order to obtain the sample, the sampler is shipped in a vertical position and removed from the shipping cask directly into the hot cell. At this time, the sampler must be placed in the horizontal position. The sample is then loaded into the mechanical extruder and removed by pushing it out from the back of the sampler with a piston. In this case, the sampler is pressed against a fixed piston, forcing the sample into the extrusion tray. If a full sample is captured, the material nearest the valve will be from a deeper part of the tank. The material near the piston is closer to the surface. The sample and any liquids are collected on a metal tray. Next, the mass of the segment and the approximate length are recorded. From this information, the gross bulk densities of the segments can be estimated until further physical properties work is performed. The sample volume is determined by measuring the length of the extruded sample using a linear unit volume of 9.85 mL/in. Figure 4-1 illustrates how the SST segment sample was extruded. Color photographs documenting the extrusions of each of the segments from tank 241-T-111 were taken and are on file at the 222-S Laboratory.

Figure 4-1. Typical Single-Shell Tank Segment Extrusion.



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Several different styles of nomenclature presently are used for distinguishing core samples, sample segments, and subsegments in the existing literature. Two major conventions are used in the documentation relating to core sampling in general. The first is designating the segment with the last two digits of the calendar year (92-) and then numbering the segments sequentially (-001, -002, etc.). This system resets itself every calendar year. The second system distinguishes the tank, core, segment (and subsegment, if necessary), with segment 1 being the at the top of the core sample and having the number increase as a function of depth in the tank so that segment 9 is at the bottom of the tank in 241-T-111. An example of this naming protocol for the second segment of the first core is 241-T-111-Core 31-Segment 2. Where no tank identification is given in this report, it is understood to mean tank 241-T-111.

4.2 TANK 241-T-111 CORE SAMPLE DESCRIPTION

The location of the risers, the dished bottom of the tank, and safety margins in the sampling protocol preclude obtaining samples from the entire waste depth in the tank. In addition, the sampling protocol establishes that segments will be calculated from the bottom up. Thus, depending on the waste depth, maximum recovery for the top segment from tank 241-T-111 is not necessarily going to be a full 48-cm (19-in) segment. However, for cores 31 and 33, sample recovery was excellent; overall recoveries were in excess of 80 percent. Segment recoveries were based on the maximum recoverable volume for the segment regardless of solid/liquid ratio. The core recoveries reported in the data package are determined based on a visual inspection of the sample length and apparent volume at the time the samples are extruded. Further study of the color photographs taken after extrusion can aid in clarifying a rough sample volume. Tables 4-1 and 4-2 present the initial measurements and observations regarding the core samples on extrusion, and an estimated range of the core recovery on a volume basis for cores 31 and 33.

Although samples for core 32 were taken from riser 2, the materials obtained at all levels appeared to be particulate-suspended in an aqueous solution, with slight traces of normal paraffin-hydrocarbon contamination observed in a few samples. These samples did not correspond to the observed conditions in the tank and were considered non-representative. The results of the core-32 sampling exercise were attributed to sampler failure, and because no acceptable samples were acquired, no assays were performed. Therefore, no results for core 32 will be reported. Valve failures were reported routinely for all three core samples at deeper positions in the tank. The full data package (McKinney et al. 1993) containing all of the assay results is available from the Hanford-Site Central Files.

General characteristics of tank 241-T-111 waste materials are as follows.

- Very little drainable liquid was associated with these samples either in the liner or in the extruder.
- Core samples generally were dark brown or black in color. The brown solids were streaked through with grey/white material.

Table 4-1. Tank 241-T-111 Core 31 Sample Description Summary.

Core number (Riser 6)	Segment	Core recovery (Vol. basis)	Total mass (g)	Comments
31	1	27%	64.0	Sampler was nearly empty; contained approximately 50 mL of black/brown low viscosity solids. Apparently homogeneous.
31	2	80-100%	182.8	Sampler was almost completely filled with solids. Again, the material is dark brown or black with a fluid or gel-like consistency, and appears to be homogeneous. A small amount of liner liquid was observed. The liquid was observed to be two phase (NPH and aqueous phases).
31	3	95-100%	162.2	Sampler was almost completely filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous.
31	4	80-100%	153.5	Sampler was almost completely filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous. The top eighth contained waste material that appeared to be more fluid than the rest of the sample. No sampler liquid or liner liquid was observed.
31	5	100%	190.9	Similar to previous observations; no sampler or liner liquid.
31	6	0%	NA	Sampler empty.
31	7	90-100%	186.4	Sampler was almost completely filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous.
31	8	100%	186.4	Similar to previous observations; no sampler or liner liquid.
31	9	100%	203.1	Sample was not homogeneous. Sample began as before (dark brown and viscous), but gradually became lighter as a function of depth. Sample was divided into two portions, a light end (133.4 g) and a dark end (69.7 g). Consistency of the sample remained the same throughout.

Table 4-2. Tank 241-T-111 Core 33 Sample Description Summary.

Core number (Riser 3)	Segment	Core recovery (Vol. basis)	Total mass (g)	Comments
33	1	100%	159.16	Sampler was full of black/brown low viscosity solids. Apparently homogeneous, with no drainable liquid.
33	2	100%	207.59	Sampler was completely filled with solids. Again, the material is dark brown or black with a viscous consistency, and appears to be homogeneous.
33	3	87-100%	167.9	Sampler was nearly filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous.
33	4	75-85%	182.05	Sampler was 75-85% filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous. The valve was observed to be open prior to extrusion. No sampler liquid or liner liquid was observed.
33	5	88%	174.3	Similar to previous observation, the valve was observed open, the sampler had approximately 88% solids, and no drainable or liner liquid was seen.
33	6	100%	217.37	Sampler was almost completely filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous.
33	7	100%	196.91	Sampler was almost completely filled with solids. The waste is dark brown with a thick, viscous consistency, and appears to be completely homogeneous.
33	8	100%	199.8	Similar to previous observations. No sampler or liner liquid.
33	9	100%	191.01	Sample was not homogeneous. Sample began as before (dark brown and viscous) but gradually became lighter in color as a function of depth, similar to core 31. Aliquots from the light and dark portions were taken for VOA and energetics analyses. Consistency of the sample remained the same throughout.

- The samples had a viscous or gel-like consistency. They were thick, relatively smooth sludges (swamp mud was the descriptive term used by the hot-cell observer). The core materials all appeared to be saturated with liquid, which did not drain.

4.3 HOLD TIME CONSIDERATIONS

All analytes have a predetermined maximum allowable holding time set by the Environmental Protection Agency (EPA 1986), during which the analysis should be completed. Completion of analysis during the maximum allowable holding time enhances the regulatory defensibility of the data. The length of the holding time varies for each analyte. For example, analyses performed on volatile and semivolatile organic compounds, many of which decompose or dissipate quickly, have shorter holding times. On the other hand, persistent analytes such as metals (except mercury), do not readily decompose or dissipate, and therefore have much longer acceptable holding times. Nearly all of the analyses of cores 31 and 33 exceeded their respective maximum allowable holding times. The only analyses that came close to meeting holding-time criteria were radiochemistry and metal analyses. Both of these analyses were completed about six months after sampling, and six months is the maximum hold time for these analyses. Although exceeding the maximum allowable holding times weakens the defensibility of the analytical results for some uses, it is anticipated that the overall effect on the analytical results for tank 241-T-111 waste relative to waste management and disposal information needs is minimal. Further discussion of holding times can be found in Winters et al. (1990a).

4.4 SAMPLE PREPARATION

Sample preparation procedures are conducted in order to optimize the recovery of each analyte of interest from the tank waste. Water digestion, acid digestion, and potassium hydroxide fusion commonly are used to extract metals and several radioisotopes from solid samples, and in some cases digestions are performed on liquid samples to improve analytical resolution. Many separations are specific to a particular analysis and are described within the corresponding analytical methods referenced in Section 4.5. In order to verify analyte recoveries resulting from separation techniques, laboratory control samples, carriers, spikes, tracers, and surrogates are analyzed concurrently with the characterization samples.

In some cases no sample preparation is necessary or desired. Direct analyses are assays performed on the sample matrix with little or no sample preparation. Several direct analyses were performed relating to the physical or energetic properties of the waste: density, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and gravimetric weight percent water.

Water leach (or water digestion) analyses are assays performed after the sample matrix has been digested in distilled/deionized water. The water is then analyzed for soluble analytes. The soluble anions are determined by ion chromatography (IC). The primary anions analyzed in this manner are fluoride, chloride, nitrate, nitrite, phosphate, and sulfate. In addition, free cyanide and pH were also analyzed from water digestion samples. Note that IC assays use a 1 to 100 sample to water digestion, where pH measurements use a 1 to 1 sample to water ratio. Selected radionuclides were measured on some of the water digestion samples to determine the type and number of water soluble radionuclides. Inductively coupled plasma-atomic emission spectroscopy (ICP) and atomic absorption (AA) spectroscopy were also performed on some of the water digestion samples. These assays were performed to determine the amount of soluble metal cations (ICP) or arsenic, mercury, or selenium (AA). In many cases, these analytes were below the detection limits in the water digestion samples, suggesting that most of the analytes are not water soluble.

Acid digestion is a preparation method in which the sample is dissolved in a mixture of nitric and hydrochloric acids. This preparation brings most of the insoluble metals into a solution with a minimum amount of dilution, and usually is best for the detection of trace and some major metals. These properties are the reason that acid digestion generally is used as the sample preparation for the homogenization tests in SW-846-based environmental sampling. However, experience with Hanford-Site tank-waste matrices has shown that acid digestion does not always provide complete solubilization, and that a more rigorous dissolution preparation, such as fusion, may be necessary to get adequate quantitation. The analyses performed on this preparation were the ICP, gamma energy analysis (GEA), and AA analysis (the AA analysis used nitric acid only). IC analysis was not performed with the acid digestion preparation solution because that method introduces additional Cl^- or NO_3^- anions, confounding the results for that sample.

Analyses that were performed on fusion-prepared samples were ICP and GEA for radionuclides. Fusion dissolution analyses are assays performed on the sample matrix after it has been fused with potassium hydroxide in a nickel crucible and dissolved in acid. This preparation dissolves the entire sample, whereas other sample preparation procedures may not completely dissolve the sample matrix. However, one significant disadvantage of fusion preparation is that large amounts of potassium hydroxide are required to bring a sample into solution. Because of this high dilution factor, trace elements are less likely to be quantified correctly, if they are detected at all. Another limitation of the preparation method is if the sample contains substantial quantities of potassium or nickel, these analytes will not be quantifiable because the procedure uses potassium hydroxide and a nickel crucible. This limitation can be overcome using alternate preparation methods if potassium or nickel are analytes critical to interpretation of the data. Elements that occur in abundance, such as major metals, or that are highly insoluble are likely to be detected better by the fusion results than by any other sample preparation.

Generally, fusion dissolution is the preferred method of analyzing radionuclide content, with the exception of ^{14}C , ^{129}I , and ^3H (tritium). However, the sample preparation specified in the test instructions for ^{14}C (water digestion) probably is not best for the high-level waste

matrices. Difficulty with dissolving the sample with a water leach, and volatility associated with a fusion preparation, potentially will bias the ^{14}C results low for both sample preparation types if they are associated with the water insoluble solid materials, and similar difficulties are encountered for the other radionuclides mentioned. However, none of these analytes are expected to be significant contributors to the radionuclide content of the waste.

Major metal components that were detected well with fusion ICP analysis for tank 241-T-111 were calcium, chromium, iron, manganese, sodium, bismuth, and lanthanum. Phosphorous, sulfur, and silicon are non-metallic analytes detected by ICP. In the case of these elements, the fusion result is the preferred method of analysis because it is believed to provide more complete dissolution of the waste, and therefore, more complete quantitation of the analytes. Comparisons of these results with the IC results can provide insight to the solubility characteristics of the waste. Some of the primary radionuclides that are measured using this sample preparation are ^{237}Np , $^{239/240}\text{Pu}$, ^{90}Sr , ^{137}Cs , and ^{99}Tc . A total alpha and total beta count were performed on the fusion dissolution samples as well.

As previously noted, chemical and radiological analyses were done largely on core composites, and in these previous characterization efforts, the core composites were built using quantities of segments based on a proportion of the total weight of sample for the core (Winters et al. 1990a, Winters et al. 1990b). This method assumed that the sample obtained is representative of what is in the tank. However, when partially filled segments are obtained, this procedure assumes that the tank does not contain any waste in this area. Incomplete recovery for a segment probably is the result of sampling problems rather than voids in the waste. The approach used in this analysis effort was to composite equal quantities of the homogenized segment material and assume that whatever is obtained in a partial segment is representative of a whole segment. Some inaccuracies may be introduced from this method because of density differences between segments. However, the inaccuracies introduced from density differences probably are small. In general, those deviations are minimal compared to the other errors inherent in core sampling and analysis. If full segments are obtained for the entire core, and the homogenization procedure is satisfactory, there will be little difference between the two approaches.

4.5 ANALYTICAL METHODS

This section briefly describes the analyses used to characterize the waste in tank 241-T-111. The analyses were split between the Westinghouse 222-S Laboratory and PNL. Several of the analytical tests performed on the composites were also performed on the segments, but on a much more limited scope. There were no free liquids from cores 31 and 33, thus there are no separate liquid core composite results. However, in March 1994, a series of liquid grab samples were taken and analyzed for compatibility considerations (Carothers et al. 1994) before the start of stabilization. These results will be presented with the water digestion results and comparisons and conclusions will be made.

4.5.1 Physical and Rheological Tests

Physical tests completed at the 222-S Laboratory included particle size analysis, TGA, DSC, specific gravity, and percent water analyses. Duplicates were performed for the percent water analyses. The physical properties measured at PNL included weight percent solids, settling behavior, and weight percent dissolved solids. Rheological testing on these samples were performed at PNL and included shear strength and shear stress as a function of shear rate. Rheological properties were measured in duplicate. Table 4-3 lists the analytical methods used for physical and rheological testing. Three segments from Core 31 (segments 2, 4, and 8) were selected to perform the full suite of rheological and physical measurements, in addition to the particle size assay done on each segment. Viscosity, settling properties, fluid behavior, and shear strength were some of the primary characteristics investigated. The samples tested for these properties were not homogenized before analysis.

Table 4-3. Analytical Methods for Physical and Rheological Testing.

Analyte	Procedure
Particle size	T044-A-01712F
Thermogravimetric analysis	LA-560-112
Differential scanning calorimetry	LA-514-113
Specific gravity	LA-510-112
Percent water	LA-564-101
Rheology	PNL-ALO-501 PNL-ALO-502
Physical properties	N/A

Scanning TGA and DSC are useful in determining the thermal stability or reactivity of a material. TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. In DSC analysis, the heat absorbed/evolved over and above the usual heat capacity of the substance is measured while the substance is exposed to a linear increase in temperature. The gravimetric weight percent water was determined by drying the sample for 12 to 24 hours in an oven at 103 to 105 °C and measuring the difference in the weight of the sample.

4.5.2 Chemical and Radionuclide Constituent Analysis

Most of the chemical and radionuclide analyses were performed at the 222-S Laboratory. The uranium and plutonium isotopic analyses, however, were performed at PNL. Duplicate analyses were performed on every tank sample. Table 4-4 lists the analytical methods used (Winters et al. 1990a, Winters et al. 1990b).

Sample Homogenization

The segment and core composite samples were homogenized using a mechanical mixer before analysis. This was done so that aliquots removed for analysis would be representative of the entire segment or core composite. Aliquots of the homogenized tank waste from core 33, segments 1, 3, 5, 7, and 9, were taken to determine the efficacy of the homogenization procedure. The samples were split into duplicates, acid digested, and assayed by ICP and GEA. This procedure is done to determine if the degree of mixing achieved by the as-planned homogenization procedure was sufficient to achieve sample homogeneity. Since the homogenization samples are evaluated concurrently or after the other core samples, the results provide only an estimate of subsampling error (or variation). They were not used in this case to ensure that homogenization was achieved before analysis. However, after review of the results, it appears that homogenization of the samples was satisfactory.

4.5.3 Organic Constituent Analyses

All organic analyses of the samples from tank 241-T-111 were performed at PNL. An EPA contract-laboratory-procedure-type organics-speciation analysis was performed on the core composites. No levels of organic compounds above the contract required quantitation limit were found in any of the samples, and they were not expected to contribute to the sample matrix. The organic analyses performed were volatile organic analyses, semi-volatile organic analyses, total organic halides, and extractable organic halides. Duplicates were performed for all of these analyses. Table 4-5 lists the analyses and procedure numbers.

At the 222-S Laboratory, the initial total organic carbon assays were done using a furnace oxidation procedure of a water digested sample. At PNL, the total organic carbon content for the solids was determined using the hot persulfate method. That method dissolves a sample in a 90 °C+ sulfuric acid solution to liberate inorganic carbon (carbonate). $K_2S_2O_8$ is then added, and organic carbon is converted to CO_2 , which is measured coulometrically. As stated in the "Executive Summary," these methods did not provide satisfactory results for the tank 241-T-111 waste samples. Later, furnace oxidation tests done at PNL gave results much higher than those from the 222-S Laboratory and more in line with the observed exothermic activity.

Table 4-4. Analytical Methods for Chemical and Radionuclide Analyses.

Analyte	Method	Procedure number
Hg	Cold vapor atomic absorption	LA-325-102
F ⁻ , Cl ⁻ , NO ₃ ⁻ , NO ₂ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻	Ion chromatography	LA-533-105
CN ⁻	Distillation/spectrometric analysis	LA-695-101 LA-695-102
U	Laser fluorimetry	LA-925-106
Total Alpha Total Beta	Proportional counting	LA-508-101
²³⁸ Pu, ^{239/240} Pu, ²⁴¹ Am	Alpha spectrometry	LA-503-156
²³⁷ Np	Alpha proportional counting	LA-933-141
Total Cations	Inductively coupled plasma	LA-505-151
⁹⁰ Sr	Beta proportional counting	LA-220-101
⁹⁹ Tc ⁷⁹ Se ¹⁴ C ³ H	Liquid scintillation	LA-438-101 LA-365-132 LA-348-104 LA-218-114
¹²⁹ I ⁵⁹ Ni	Low energy gamma analysis	LA-378-104 PNL-ALO-464
⁶³ Ni	Liquid scintillation	PNL-ALO-474
¹⁵⁴ Eu, ¹⁵⁵ Eu, ²⁴¹ Am, ¹³⁷ Cs, ⁶⁰ Co	Gamma energy analysis	LA-548-121
NO ₂ ⁻	Spectrophotometry	LA-645-001
H ⁺	pH	LA-212-103
As Se	Graphite furnace atomic absorption	PNL-ALO-214 PNL-ALO-215
Pu Isotopic	Fusion mass spectrometry	PNL-ALO-423 PNL-MA-597
U Isotopic	Mass spectrometry uranium laser	PNL-MA-597 PNL-ALO-445
TOC	Total organic carbon	LA-344-105 PNL-ALO-380 PNL-ALO-381
CO ₃ /C	Total inorganic carbon	LA-622-102

Table 4-5. Analytical Methods For Organic Analytes.

Analysis	Method	Procedure Number
Volatile organic analysis	Gas chromatography/mass spectrometry	PNL-ALO-335
Semi-volatile organic analysis	Gas chromatography/mass spectrometry	PNL-ALO-345
Extractable organic halides	Microcoulometric titration	PNL-ALO-320.2
Total organic halides	Microcoulometric titration	PNL-ALO-321

4.5.4 Segment-Level Analyses

The objectives of segment-level analyses are to provide (1) information as a function of depth pertaining to the overall waste energetics (water content and chemical reactivity) and (2) the particle size distribution and other general rheological information. To accomplish these goals, the limited suite of analyses listed in Table 4-6 were performed on each homogenized segment. These analyses were conducted using the analytical procedures identified in Tables I5-1 and I5-2 of WHC-EP-0210, Rev. 3 (Hill et al. 1991). In addition, where appropriate, the information obtained from the segment-level homogenization tests will be used to enhance the interpretation of the data.

Table 4-6. Segment-Level Analysis.

Direct	Acid dissolution*
Thermogravimetric analysis differential scanning calorimetry Wt% H ₂ O particle size**	Inductively coupled plasma (metals) gamma energy analysis (¹³⁷ Cs) total alpha

*Acid dissolution assays were performed on the homogenization test segments.

**Particle size was done on non-homogenized segment material.

5.0 ANALYTICAL RESULTS: TANK 241-T-111

5.1 CHEMICAL ANALYSES

5.1.1 Analytical Results: Inductively Coupled Plasma-Atomic Emission Spectroscopy

Online inter-element corrections were performed for matrix interferences. The ICP has a built-in correction capability to adjust for moderate matrix interferences; however, there may be performance degradation on samples containing weight-percent quantities of iron, aluminum, or uranium. Corrections were made for moderate levels of aluminum, calcium, chromium, and magnesium in the samples. Corrections were made for high iron concentrations as well. Process or preparation blank values have not been subtracted from the results. In the water digestion and liquid grab sample assays, the single most prevalent element is sodium by at least an order of magnitude. Relative percent differences (RPDs) for water digestion results were high for some analytes (i.e. above the 20 percent acceptance criteria), but there was no consistent trend observed between cores 31 and 33.

In the fusion assays, some elements can appear to be at high concentrations because of the large dilution factors required for fusion samples. These high dilution factors propagate errors. Those analytes actually may be present only in concentrations marginally above the detection limit. For several analytes, higher quantitation was found in the acid digestion results. For purposes of determining inventories and making comparisons, the highest reliable average analytical result will be used between acid and fusion preparations.

In reviewing the data, a subtle bias or gradient was observed between the results for cores 31 and 33, with core 31 having slightly higher values overall than core 33. Although not readily discernable among the first tier analytes (sodium, bismuth, iron, and phosphorus), the difference between cores 31 and 33 is more evident in the second tier analytes (aluminum, lead, and magnesium). The RPDs between the individual core composite samples and their replicates were small, suggesting that the gradient is real and not an analytical artifact, however, this observation could be the result of the compositing procedure. There were nine segments used in the core 33 composites, but only eight segments in the core 31 composites (segment 6 was absent in core 31). Furthermore, sample variability may contribute or be wholly responsible for the observed difference.

RPDs for most elements were within the 20-percent acceptance criteria for acid and fusion results, and generally were less than 10 percent for the major analytes. Potential sample contamination for boron and silicon exists because of the caustic nature of the samples and the glass vials used to store the samples in the laboratory; however, silicon routinely demonstrated a low bias. Calcium and magnesium for blank and spike recovery results may have been biased high by the powder used on the analysts' gloves when performing the assays. With the small sample sizes used in the assay, even trace amounts of powder have

the potential to impact the analyses. Low spike recoveries were noted for several analytes for differing reasons. Silver recoveries are commonly low because of the precipitation of silver chloride in the sample digestion. Poor spike recoveries of iron, magnesium, and calcium accompany high preparation blank results, but the overall correlation is poor. Spike failures frequently are noted for major elements when the spike concentration is insignificant compared to the analyte concentration in the waste matrix. Spike and standard results outside the acceptance criteria for these analytes do not necessarily invalidate the sample results for the ICP in general, or for those analytes in particular. Individual analyte failures need to be evaluated on a case by case basis. All of these behaviors could affect, and are considered in the interpretation of the results.

The detection limit for each analyte is provided for comparison with the results to aid in interpretation. All ICP analytes are reported in the data tables; however, those consistently contributing significant (i.e. generally greater than approximately 0.2 weight percent) amounts to the composition of the waste matrix generally are relevant to bulk characterization. The fusion/acid ratio, which can be compared in the table, for most analytes indicates near total dissolution for the acid digestion assay. Average values for the analytes are reported to three significant figures. The full range of ICP analytical results can be found in the full data packages (McKinney et al. 1993). All reported concentration values are based on grams of wet sample, unless otherwise specified. Table 5-1 provides ICP analyte concentration information on the core composites as a function of the sample preparation. Table 5-2 provides ICP analyte concentrations as a function of depth for Core 33. Table 5-3 compares the water digestion core composite ICP values with the results from the grab sample.

Core 31

The most significant analytes measured by the water digestion of the core composite were sodium and phosphorous (probably as a soluble phosphate), and sulfur (probably as a soluble sulfate). Much smaller amounts of iron, chromium, and silicon also were measured. RPDs were elevated for these samples (between 10 and 20 percent), but were generally within the 20-percent acceptance criteria. For analytes with results outside the acceptance criteria, no clear trend between the two composites can be established. The water digestion result for core 31, composite 1 had several analytes with high RPDs, many of which are significant contributors to the waste: iron, lead, manganese, silver, bismuth, lanthanum, and strontium. Core 31, composite 2 had few analytes with results outside the acceptance criteria: barium, calcium, lead, and vanadium. All of the analytes noted are largely insoluble and that characteristic probably is contributing to the observed variability.

The results from the acid digestion preparation of the core composite samples had sodium, phosphorous, sulfur, calcium, chromium, iron, manganese, bismuth, and lanthanum as major analytes. RPDs are very good in general for most of the major analytes (generally between 5 and 10 percent), well within the 20 percent acceptance criteria. Analytes outside the acceptance criteria for core 31, composite 1 were antimony and boron (56 percent and 27 percent, respectively). However, neither of those analytes are substantial contributors to the waste matrix, and for analytes near the detection limit, reproducibility is not expected.

Table 5-1. ICP Cations. (4 pages)

Analyte	Acid-water digestion	Fusion digestion	Prep type	Core 31 comp. 1	Core 31 comp. 2	Core 33 comp. 1	Core 33 comp. 2
	Detection limit	Detection limit		Average concentration	Average concentration	Average concentration	Average concentration
	($\mu\text{g/g}$)	($\mu\text{g/g}$)		($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)
Al	2.4	12.0	Water	6.71	10.3	15.5	11.0
			Acid	584	705	472	405
			Fusion	644	693	484	459
Sb	17.7	85.5	Water	17.7	17.7	17.7	17.7
			Acid	30.4	36.5	35.9	22.6
			Fusion	88.6	88.3	109	88.4
As	3.0		Water	3.0	3.0	2.99	3.0
			Acid	3.15	2.92	3.45	3.06
			GFAA ¹	3.3	3.3	3.3	3.2
	3.3	15.0	Fusion	15.0	15.0	15.0	15.0
Ba	.3	1.5	Water	0.305	0.416	0.547	0.516
			Acid	57.0	64.9	66.8	87.3
			Fusion	58.8	60.6	65.4	73.7
Be	0.1	0.5	Water	0.10	0.10	0.10	0.10
			Acid	0.105	0.097	0.117	0.104
			Fusion	0.501	0.499	0.499	0.499
Cd	.4	2.0	Water	0.40	0.40	0.4	0.4
			Acid	7.22	7.86	4.4	3.72
			Fusion	8.25	10.7	6.42	7.17
Ca	4.4	22.0	Water	50.8	61.6	66.4	67.5
			Acid	2,200	2,480	1,490	1,350
			Fusion	2,760	2,660	2,220	2,050
Cr	0.9	4.5	Water	209	229	224	211
			Acid	1,860	1,840	2,060	2,140
			Fusion	1,890	1,700	1,790	1,820

¹GFAA: Graphite furnace atomic absorption

Table 5-1. ICP Cations. (4 pages)

Analyte	Acid-water digestion	Fusion digestion	Prep type	Core 31 comp. 1	Core 31 comp. 2	Core 33 comp. 1	Core 33 comp. 2
	Detection limit	Detection limit		Average concentration	Average concentration	Average concentration	Average concentration
	($\mu\text{g/g}$)	($\mu\text{g/g}$)		($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)
Co	0.8		Water	0.8	0.825	0.821	0.825
			Acid	3.4	7.16	3.13	2.91
		4.0	Fusion	10.1	10.8	13.3	11.8
Cu	.4		Water	0.4	0.4	0.4	0.4
			Acid	25.2	79.6	16.4	13.0
		2.0	Fusion	36.3	34.2	22.1	24.6
Fe	1.0		Water	79.6	140	132	159
			Acid	19,200	20,000	17,500	17,300
		5.0	Fusion	20,500	19,600	15,900	16,100
Pb	6.2		Water	7.05	7.93	6.24	6.19
			Acid	475	543	201	168
		31.0	Fusion	440	484	267	269
Mg	0.3		Water	2.94	3.95	3.84	3.83
			Acid	435	479	305	290
		1.5	Fusion	438	443	268	272
Mn	0.2		Water	14.7	25.1	25.4	33.8
			Acid	6,190	6,140	6,710	6,280
		1.0	Fusion	6,380	5,940	6,220	6,590
Ni	1.7		Water	1.7	1.7	1.7	1.7
			Acid	151	157	110	108
		8.5	Fusion	NR	NR	NR	NR
K	11.2		Water	734	783	712	648
			Acid	1,100	1,210	1,210	1,020
		56.0	Fusion	NR	NR	NR	NR
Se	7.6		Water	7.6	8.01	7.58	7.59
			Acid	7.98	7.4	10.3	7.79
	1.5		GFAA	1.5	1.5	1.5	1.5
		38.0	Fusion	38.0	37.9	38.0	38.0

Table 5-1. ICP Cations. (4 pages)

Analyte	Acid-water digestion	Fusion digestion	Prep type	Core 31 comp. 1	Core 31 comp. 2	Core 33 comp. 1	Core 33 comp. 2
	Detection limit	Detection limit		Average concentration	Average concentration	Average concentration	Average concentration
	($\mu\text{g/g}$)	($\mu\text{g/g}$)		($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)
Ag	0.5		Water	0.639	1.16	0.553	0.499
			Acid	203	227	44.3	30
		2.5	Fusion	214	221	39.5	37.1
Na	3.1		Water	34,000	35,000	30,800	32,000
			Acid	37,600	38,700	35,000	36,300
		15.5	Fusion	39,800	39,000	33,900	35,200
V	0.5		Water	0.5	0.707	0.5	0.8
			Acid	12.7	21.4	13.9	9.99
		2.5	Fusion	12.1	16.5	15.3	14.7
Zn	0.3		Water	0.3	0.3	0.3	0.3
			Acid	79.4	101	44.2	35
		1.5	Fusion	104	106	105	110
Bi	7.5		Water	115	191	231	270
			Acid	23,600	23,300	28,500	28,400
		37.5	Fusion	20,900	20,100	26,500	26,700
B	0.6		Water	3.31	3.19	5.54	4.25
			Acid	27.1	23.4	29.4	32.2
		3.0	Fusion	3.0	2.99	4.84	4.84
Ce	10.1		Water	10.1	10.1	10.1	10.1
			Acid	32.6	28.6	37.8	35.8
		50.5	Fusion	50.6	50.4	50.4	50.4
La	1.4		Water	6.02	8.52	13.8	15.8
			Acid	3,720	3,620	4,640	4,890
		7.0	Fusion	3,690	3,410	4,510	4,810
P	5.8		Water	5,760	5,960	5,300	5,700
			Acid	10,100	9,960	9,860	11,300
		29	Fusion	11,600	11,100	9,070	9,910
Si	1.3		Water	438	560	669	620
			Acid	482	471	528	394
		6.5	Fusion	5,960	5,840	5,460	5,410

Table 5-1. ICP Cations. (4 pages)

Analyte	Acid-water digestion	Fusion digestion	Prep type	Core 31 comp. 1	Core 31 comp. 2	Core 33 comp. 1	Core 33 comp. 2
	Detection limit	Detection limit		Average concentration	Average concentration	Average concentration	Average concentration
	($\mu\text{g/g}$)	($\mu\text{g/g}$)		($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)
Sr	0.3		Water	1.21	2.13	2.18	2.34
			Acid	282	280	305	334
		1.5	Fusion	303	280	291	317
S	2.7		Water	1,190	1,200	1,060	1,140
			Acid	1,230	1,260	1,140	1,220
		13.5	Fusion	1,350	1,310	1,080	1,160
Sn	1.6		Water	1.6	1.6	1.6	1.6
			Acid	4.21	2.44	1.81	1.61
		8.0	Fusion	8.01	7.98	7.99	7.99
Ti	0.4		Water	0.4	0.4	0.4	0.4
			Acid	29.4	33	8.9	6.46
		2.0	Fusion	72.9	72.4	22.3	24.1
Zr	NR		Water	0.8	0.799	0.798	0.798
			Acid	0.84	0.778	0.913	0.816
		NR	Fusion	4.00	3.99	4.00	4.00
Hg	0.125	NR	CVAA ²	1.59	1.83	1.20	1.08

NR = Not reported

Table 5-2. Tank 241-T-111 Core 33 ICP Selected Analyte Trending as a Function of Depth (Acid prep on segments).

Segment	Ca ($\mu\text{g/g}$)	Cr ($\mu\text{g/g}$)	Fe ($\mu\text{g/g}$)	Mn ($\mu\text{g/g}$)	Ni ($\mu\text{g/g}$)	Na ($\mu\text{g/g}$)	Bi ($\mu\text{g/g}$)	La ($\mu\text{g/g}$)	P ($\mu\text{g/g}$)	Si ($\mu\text{g/g}$)	S ($\mu\text{g/g}$)
1	4,800	490	17,300	23,900	240	22,400	760	3	4,400	380	800
3	1,100	1,200	11,700	3,800	70	25,800	24,200	3,900	4,800	430	720
5	1,300	2,000	16,100	2,800	90	32,100	33,400	5,100	9,100	400	1,100
7	1,100	2,500	18,100	4,200	70	36,000	34,700	4,900	12,200	450	1,200
9	950	2,000	16,600	4,600	90	40,700	24,100	4,200	15,300	450	1,400

²CVAA: Cold vapor atomic absorption

Table 5-3. Grab Sample/Water Digestion Data--ICP Average Values.

Analyte	Grab sample avg. concentration ($\mu\text{g/g}$ sample)	Core 31, comp. 1 ($\mu\text{g/g}$ sample)	Core 31, comp. 2 ($\mu\text{g/g}$ sample)	Core 33, comp. 1 ($\mu\text{g/g}$ sample)	Core 33, comp. 2 ($\mu\text{g/g}$ sample)
Ca	< DL	51	62	66	68
Cr	230	210	230	220	210
Fe	< DL	80	140	130	160
Ni	< DL	15	25	25	34
Na	24,800	34,000	35,000	30,800	32,000
Bi	< DL	120	190	230	270
La	< DL	6	9	14	16
P	3,200	5,800	6,000	5,300	5,700
Si	60	440	560	670	620
S	750	1,200	1,200	1,100	1,100

< DL = below detection limit

This behavior is true in general for analytes with these characteristics. The silicon RPD is marginal at 19.26 percent, but acceptable. For core 31, composite 2, cobalt, copper, silicon, and tin were outside the acceptance criteria. Again, cobalt, copper, and tin were not significant contributors to the waste, and thus have little influence on the interpretation of the data. The only clear trend for this assay is for silicon. The variation observed for silicon was attributed to the solubility of the waste matrix (i.e. the waste is not completely solubilized by the acid), and therefore, the fusion results will be reviewed to quantitate silicon. Based on a ratio of the acid and water digestion results, most of the analytes are not water soluble, except as noted earlier, however, approximately 10 percent of the chromium is water soluble.

The results from the fusion preparation core composite had sodium, phosphorous, bismuth, manganese, lanthanum, silicon, iron, calcium, and chromium as major analytes. Nickel was detected in substantial quantities, but the results are considered unreliable because of sample contamination (the method uses a nickel crucible to perform the fusion), and nickel was not detected in significant quantity in the acid digestion assay. RPDs were quite low, generally less than eight percent for most analytes, demonstrating excellent agreement. The only analyte with an RPD outside the acceptance criteria for core 31, composite 1 was cadmium, which is not a large contributor to the waste and is near the detection limit. The only analytes with excessive RPDs for core 31, composite 2 were cadmium and nickel. Both of these analytes have problems associated with them that make these results suspect for this test method (proximity to the detection limit for cadmium and cross-contamination from the

crucible for nickel). Comparisons with the acid digestion results indicate that the samples were well dissolved by the acid preparation, but in some cases, and with silicon especially, the fusion dissolution was necessary to obtain reliable, quantitative results for the analyte.

Core 33

Sodium, phosphorus (probably as a soluble phosphate), and sulfur (probably as a soluble sulfate) were the most significant analytes measured by the water digestion of the core composite. Much smaller amounts of iron, chromium, and silicon were the other main analytes. RPDs were elevated for these samples (between 10 and 20 percent), but generally were within the 20 percent acceptance criteria. For analytes with results outside the acceptance criteria, no clear trend between the two composites or between cores can be established firmly, but there was some correspondence between the cores and their respective composites (i.e. there was some agreement between cores 31 and 33 composite 1, etc.), but the connection was quite tenuous. The water digestion result for core 33, composite 1 had several analytes with high RPDs, many of which are significant contributors to the waste. These analytes are aluminum, barium, iron, manganese, bismuth, and lanthanum. For core 33, composite 2, only calcium and vanadium were outside the acceptance criteria. The degree of variability observed for this particular sample preparation is not unexpected. Most of these analytes probably are in a form that is not readily water soluble and, depending on the sample matrix exposure to the solution media, substantial differences may be observed.

The results from the acid digestion preparation of the core composite samples are quite similar to core 31. They had sodium, phosphorous, sulfur, calcium, chromium, iron, manganese, bismuth, and lanthanum as major analytes, the RPDs decline significantly in comparison to the water digestion results, both in the number of analytes outside the acceptance criteria and the overall magnitude of the RPDs themselves. The RPDs are very good in general for most of the major analytes (between 5 percent and 10 percent), well within the 20 percent acceptance criteria. Analytes outside the acceptance criteria for core 33, composite 1 were antimony and selenium (88.5 percent and 33.0 percent, respectively). For core 33, composite 2, antimony and silicon were outside the acceptance criteria (31.2 percent and 48.7 percent, respectively). The results for antimony and selenium are not surprising. They are not far above their respective detection limits and neither is a substantial contributor to the waste matrix. The silicon RPD result is not unexpected, because the waste has solubility properties that make it resistant to acid digestion, making this assay marginal at best. Based on a ratio of the acid and water digestion results, most of the analytes are not water soluble, except as noted earlier; however, approximately 10 percent of the chromium is water soluble. In this case, for several analytes, some consistency is seen between composite replicates and cores. As noted earlier, core 33 sample results are, in several cases, about 25 percent or more lower than core 31. Significant changes in analyte concentration were observed in the ICP acid digestion results as a function of depth for core 33. The results are shown in Table 5-2. The changes observed for some analytes are notable, ranging from factors of 3 and 4 for calcium, chromium, and phosphorous to orders of magnitude for manganese, bismuth, and lanthanum. These swings

in concentration suggest the waste is heterogenous on a tank-wide scale and that the waste in the tank is layered.

Properties and major analytes for the fusion digestion results are similar to core 31, however the differences observed between the analytical results of cores 31 and 33 become much less consistent with this group of assay results. RPDs were generally low, less than 12 percent, with most analyte results substantially less than that. Antimony, calcium, and boron had RPDs outside the acceptance criteria for core 33, composite 1. In core 33, composite 2, cobalt and nickel, which are not large contributors to the waste, had RPDs outside the acceptance level and antimony, boron, and cobalt are near their respective detection limits. Several of these analytes have problems associated with them that make these results suspect for this test method. However, the variation seen for calcium is not expected and there is no reason for its behavior. In this case, no consistency was observed between composites or cores, except for nickel in cores 31 and 33, composite 2, and the nickel results for this test method are suspect.

5.1.2 Analytical Results--Anion Assays

Core 31 Ion Chromatography Results:

Major anions detected are NO_3^- and PO_4^{3-} , with smaller, but still substantial amounts of F^- and SO_4^{2-} . Nitrite and chloride are minor contributors to the waste. Indeed, the IC results for nitrite are considered an estimated result; however, the magnitude and range of concentration is confirmed by a spectrophotometric analysis. All RPDs for quantitated results are well within the 20 percent acceptance criteria, and generally are less than 10 percent. Comparisons of phosphorous and sulfur water-digestion ICP results with ion-chromatography results for PO_4^{3-} and SO_4^{2-} give good agreement (RPDs are less than 10 percent) and show that phosphorous is about 48 to 56 percent soluble (as phosphate) and sulfur is about 90 to 100 percent soluble (as sulfate). Table 5-4 illustrates the comparison between the water digestion ICP and IC results, and the relationship to the total amount of phosphorous and sulfur in the matrix. Table 5-5 presents the summary results for the IC analytes and other anions.

Core 33 Ion Chromatography Results:

Major analytes were the same as those found in core 31. RPDs for the minor analytes, F^- , Cl^- , and NO_2^- , were much higher in composite 1 than in composite 2, or in either of core 31's composites, exceeding 15 percent, but within the 20 percent acceptance criteria. Core 33, composite 2 had superior RPDs, all were less than 8 percent. Comparison of the phosphorous and sulfur water-digestion ICP results with IC for PO_4^{3-} and SO_4^{2-} gives good

Table 5-4. Comparison of Ion Chromatography and Water Digestion ICP Results for Selected Analytes.

Sample ID	PO ₄ ³⁻ Concentration (μg/g)		PO ₄ ³⁻ RPD	SO ₄ ²⁻ Concentration (μg/g)		SO ₄ ²⁻ RPD	PO ₄ ³⁻ Solubility*		SO ₄ ²⁻ Solubility*	
	(IC)	(ICP.w)		(IC)	(ICP.w)		(IC)	(ICP.w)	(IC)	(ICP.w)
Core 31-C1	16,200	17,300	6.7	3,690	3,570	-3.3	50.8	54.2	100.4	97.1
Core 31-C2	17,400	17,900	2.8	3,740	3,600	-3.7	54.5	56.1	101.8	97.9
Core 33-C1	13,600	15,900	16.9	3,290	3,420	-3.3	42.6	49.8	89.5	93.1
Core 33-C2	15,100	17,100	13.2	3,470	3,660	-1.4	47.3	53.6	94.4	99.6

*Solubility is a ratio of the IC/ICP.w result to 31,900 μg/g PO₄³⁻ and 3,675 μg/g SO₄²⁻, the tank average of the converted fusion results

ICP.w = Notation for ICP water digestion result

Table 5-5. Anion Results--Composite Data (water leach).

Analyte	Detection limit (μg/g)	Core 31 (μg/g)		Core 33 (μg/g)		Grab sample (μg/g)
		Composite 1	Composite 2	Composite 1	Composite 2	Average
NO ₃ ⁻	100	44,300	43,800	36,900	40,100	30,300
PO ₄ ³⁻	100	16,200	17,400	13,600	15,100	8,400
SO ₄ ²⁻	100	3,690	3,740	3,290	3,470	2,900
Cl ⁻	10	470	497	401	432	490
F ⁻	10	3,090	3,130	1,370	1,630	2,100
NO ₂ ⁻	50	952	525	878	817	1,320 (IC)
TOC [*]	500	3,490	3,990	2,000	3,000	420
TIC [*]	500	650	824	823	950	670
Free OH ⁻	NM	NM	NM	NM	NM	3,000
pH [*]	NA	10.18	9.93	10.05	9.77	11.65
NH ₃ /NH ₄ ⁺	4,500	< DL	< DL	< DL	< DL	400

Table 5-5. Anion Results--Composite Data (water leach).

Analyte	Detection limit ($\mu\text{g/g}$)	Core 31 ($\mu\text{g/g}$)		Core 33 ($\mu\text{g/g}$)		Grab sample ($\mu\text{g/g}$)
		Composite 1	Composite 2	Composite 1	Composite 2	Average
Direct Cyanide ¹	5	< DL	< DL	< DL	< DL	< DL

NM = No measurement NA = Not applicable

< DL = Below detection limit

Note: All IC results are obtained from a water leach preparation and are reported on a wet basis.

¹Direct Cyanide, $\text{NH}_3/\text{NH}_4^+$, pH, total organic carbon (TOC), and total inorganic carbon (TIC) are not IC analyses, but are grouped with the anions for convenience. NO_2^- results reported are from spectrophotometric methods. At the 222-S Laboratory, TOC and TIC assays were done on water digested samples. When 325 performed segment level assays for these analytes, they did them on direct solid samples.

agreement, however, phosphorous shows a higher, but still acceptable variability, with RPDs of 13 to 17 percent, than sulfur as SO_4^{2-} , with RPDs of -1.4 to -3.3. Phosphorous is approximately 50 percent soluble as phosphate, whereas sulfur as sulfate is almost entirely soluble. Table 5-4 illustrates the comparison between the water digestion ICP and IC results, and the relationship to the total amount of phosphorous and sulfur in the bulk tank matrix. Note the ICP results are converted to phosphate and sulfate for comparison.

Core 31 and 33 Additional Anion Results:

- Nitrite, as determined from spectrophotometry, has good agreement between composites and acceptable RPDs. Concentrations between cores are observed to agree well also.
- The pH of the solids is measured using a water dilution of a solids aliquot. The pH of the mixture is measured and reported. The results for tank 241-T-111 waste from this method are consistent, ranging from 9.8 to 10.5, but are of limited utility because the sample preparation and assays only marginally represent the conditions in the tank. The pH as determined from a grab sample taken in 1994 (see Appendix C) is between 11.5 and 11.8, and is considered more reliable and more representative of the waste tank conditions.

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- Ammonia/ammonium results for the solids were all below the detection limit for the method. This is not unexpected. Over time, ammonia probably was slowly released to the vapor space and removed by passive ventilation. A small amount of this analyte remains in the liquids, but the liquid is being removed.
 - Total organic carbon (TOC) results indicate a substantial difference between cores 31 and 33 (core 31's TOC concentration is 33 percent higher than core 33's), but results for the individual core composites are very consistent. Core 31 composite 1 and composite 2 RPDs are negligible. Core 33, composite 1 and composite 2 RPDs are low, well within the acceptance criteria, but noticeable.
 - Total inorganic carbon (TIC) results were a factor of three to four times lower than the TOC results and were near the detection limit. Therefore, they are not considered wholly reliable, however the results did provide acceptable reproducibility. TOC results are also considered questionable, especially when contrasted with the calorimetry results from segment-level data from both cores (segments 1-3 particularly), and a furnace oxidation value of 4.1 weight percent (dry), for core 33, segment 2 (Baldwin 1994).

The gradient observed in the ICP results remains noticeable for the anions, especially for TOC and F⁻, but the overall effect is much less pronounced. This effect is expected and ascribed to the nature of the assay (i.e. water digestion assay results for soluble analytes in a mostly water matrix are expected to be reasonably homogeneous). TOC and F⁻ probably are present in insoluble forms, and thus are not as amenable to detection and are more likely to exhibit variations in concentration.

5.2 ANALYTICAL RESULTS--RADIOCHEMISTRY

5.2.1 Radiochemistry Assays--General Comments

Analyses appear to be consistent. Total beta measurements calculated using ⁶⁰Co detector efficiencies are largely in agreement with the sum of the major beta emitters, ⁹⁰SrY and ¹³⁷Cs [i.e. beta emission values must be adjusted by 1.42 and 1.51, respectively, the ratio of ⁹⁰SrY and ¹³⁷Cs to ⁶⁰Co detector efficiencies (Winters 1991)]. However, the total alpha values show only marginal agreement with the sum of the neptunium, plutonium, and americium/curium values. This behavior was attributed to the low activity of the samples and a recently discovered equipment problem. Technicians at the 222-S Laboratory identified an electronic problem with their alpha detection system involving the degree of discrimination between beta and alpha emissions. These two conditions are thought to contribute to the observed high bias of the total alpha measurements. Detection and quantitation of ¹³⁷Cs and most other radionuclides was based on the presumed ability of the fusion sample preparation to completely dissolve the waste. Water preparation samples were

done on selected analytes as directed in the test plan. GEA measurements show good agreement with alpha energy analysis for ^{241}Am . GEA analytical values are not corrected to account for decay, however most of the radionuclides present are long-lived enough and low enough in concentration so that any correction at this point would be modest.

5.2.2 Gamma Energy Analysis Results

The GEA data from the replicate samples of the core composites for cores 31 and 33 prepared by caustic fusion agree reasonably well with each other (RPDs generally were within 10 percent). However, there is a discernable gradient between cores 31 and 33 for ^{137}Cs . This follows the general pattern observed for several other analytes, but in this case ^{241}Am does not appear to be affected. Review of the segment-level homogenization data for core 33 indicates that in general, the ^{137}Cs content is lowest in the bottom segments and highest in the upper segments, increasing in concentration from bottom to top. This behavior indicates that the ^{137}Cs is concentrated in the upper portion of the tank. However, the overall radionuclide content of tank 241-T-111 is extremely low. The ^{154}Eu and ^{155}Eu content is above the detection limits only in segments 1 and 3, suggesting that these isotopes are in the upper portions of cores 31 and 33. However because of the limited segment level data, too many conclusions should not be drawn from these observations. Grab sample results for ^{137}Cs is more than a factor of four lower than those obtained from fusion assays, indicating that the radionuclides, ^{137}Cs in particular, are very insoluble. Agreement between the same top and bottom aliquots in the homogenization check samples prepared by acid digestion were very good for analytes above the detection limit, with RPDs generally less than 10 percent and usually much less than that, indicating sample homogeneity and/or relatively complete dissolution.

5.2.3 Total Alpha Analysis and Uranium Assay

Total alpha, plutonium, ^{237}Np , ^{241}Am , and ^{244}Cm analyses were performed on the fusion prepared samples of the core composites and selected assays were done on the liquid grab sample. Total alpha measurements were also performed on the homogenization check samples from segments 1, 3, 5, 7, and 9 from core 33. The total alpha activity was determined by drying a small aliquot of prepared sample on a counting plate and assayed with an alpha proportional counter. The plutonium and americium fractions were separated by solvent extraction or ion exchange and similarly counted.

The plutonium analyses are reported as total alpha $^{239/240}\text{Pu}$. The process blank was two to three orders of magnitude lower than the samples, indicating little contamination occurred during sample preparation. The total alpha concentration frequently tends to be somewhat lower than the sum of the individual alpha emitters. The difference is probably caused by absorption by the salt residue on the counting mounts. However, in this case, substantial differences are observed between the total alpha measurements and the sum of the individual alpha emitters. The total alpha emissions are believed to be lower than the measurement

indicates because of a bias caused by beta emissions confounding the detector. The activity of the samples is so low that the offset used to discriminate between alpha and beta plateaus was not sufficient to provide accurate readings. Furthermore, analyses from PNL indicate a potential low bias for plutonium in the samples. The degree of discrepancy ranges from over a factor of two to nearly a factor of five. This degree of disagreement warrants further attention. Isotope content was determined by thermal-ionization mass spectroscopy. Little variation in the plutonium isotopic composition was observed between cores. Total alpha measurements vary widely as a function of depth, but exhibit a general decreasing trend the deeper the samples are in the waste. Segment 5 was an exception to this trend. The anomalous total alpha reading in segment 5 may be indicative of a process upset or change in waste management operations, such as the 5-6 waste that Anderson (1990) notes was co-mingled with the 2C waste in 1952.

Uranium measurements were obtained from laser fluorimetry of the fusion-prepared sample from the two core composites and their replicates. The assays show good agreement between duplicates for each individual core composite, but there is not good agreement between the core 31 and 33 replicates. Additionally, the gradient observed in the other assays is not evident here. Furthermore, analyses from PNL indicate a potential low bias for uranium in the samples. The degree of discrepancy is nearly a factor of two for corresponding samples, and thus warrants further study. Differences in concentration as a function of the water content of the samples is not deemed to be an issue because the additional characterization work done in resolving the energetics question indicates that little or no water is lost while the samples are stored. Time lag is also not considered relevant, since the difference in time between the two assays was small in comparison to the half-lives involved. No general trend of the uranium concentration as a function of depth can be established because there is no segment-level data for this analyte.

5.2.4 Total Beta

Total beta, ^{90}Sr , and ^{99}Tc analyses were performed on the liquid grab sample and fusion prepared samples of the core composites. The total beta activity was determined by drying a small aliquot of prepared sample on a planchet and assaying it with a beta proportional counter. The ^{90}Sr fraction was separated by solvent extraction or ion exchange and counted. The ^{99}Tc fraction was separated similarly, but assayed using liquid scintillation. There generally is good agreement (RPDs were less than 10 percent) between duplicates, and preparation blank beta activities are orders of magnitude lower than the levels found in the samples, again indicating little contamination from preparation in the hot cell. Most of the beta activity in the tank samples is from ^{90}Sr and ^{137}Cs . There is also a trace of ^{99}Tc . The ^{90}Sr , ^{99}Tc , and ^{137}Cs data are consistent between the fusion core composites and their replicates, but in this case the gradient between cores 31 and 33 is observed for ^{90}Sr and ^{137}Cs . There is no data to determine if ^{90}Sr content varies as a function of depth.

Tables 5-6, 5-7, 5-8, and 5-9 show the average radionuclide concentrations found in the core composite samples. Table 5-10 shows average fission product concentration and total alpha concentrations as a function of depth.

Table 5-6. Consolidated Radionuclide Concentration Results for 241-T-111.

Analyte method	Detection limit	Core 31, composite 1	Core 31, composite 2	Core 33, composite 1	Core 33, composite 2
GEA analytes	($\mu\text{Ci/g}$)	($\mu\text{Ci/g}$)	($\mu\text{Ci/g}$)	($\mu\text{Ci/g}$)	($\mu\text{Ci/g}$)
$^{60}\text{Co.f}$	(DL = $4.07\text{E-}04$)	< DL	< DL	< DL	< DL
$^{137}\text{Cs.f}$	(DL = $3.70\text{E-}04$)	0.211	0.237	0.114	0.103
$^{154}\text{Eu.f}$	(DL = $1.20\text{E-}03$)	0.00108	0.00324	< DL	< DL
$^{155}\text{Eu.f}$	(DL = $5.95\text{E-}04$)	< DL	< DL	0.00307	< DL
$^{241}\text{Am.f}$	(DL = $1.14\text{E-}04$)	0.0459	0.0409	0.0387	0.0443
Beta emitters					
Total beta.f	(DL = $9.35\text{E-}02$)	20.6	21.5	9.59	8.83
Total beta	Calculated	20.7	21.5	10.5	9.9
$^3\text{H.w}$	(DL = $3.15\text{E-}04$)	< DL	< DL	< DL	< DL
$^{14}\text{C.w}$	(DL = $2.25\text{E-}04$)	< DL	< DL	< DL	< DL
$^{59}\text{Ni.a}$	(DL = $4.00\text{E-}06$)	$8.3\text{E-}05$	$3.33\text{E-}05$	$4.44\text{E-}05$	$4.07\text{E-}05$
$^{63}\text{Ni.a}$	(DL = $4.00\text{E-}06$)	0.0093	0.00358	0.00545	0.00459
$^{79}\text{Se.f}$	(DL = $1.40\text{E-}04$)	< DL	< DL	< DL	< DL
$^{90}\text{Sr.f}$	(DL = $2.15\text{E-}03$)	7.16	7.43	3.65	3.43
$^{99}\text{Tc.f}$	(DL = $4.60\text{E-}03$)	0.00514	0.00473	0.0114	0.0104
$^{129}\text{I.f}$	(DL = $6.40\text{E-}03$)	< DL	< DL	< DL	< DL

Table 5-6. Consolidated Radionuclide Concentration Results for 241-T-111.

Analyte method	Detection limit	Core 31, composite 1	Core 31, composite 2	Core 33, composite 1	Core 33, composite 2
Uranium and transuranics					
Total U.f ($\mu\text{g/g}$) (325)	(DL = 3.40E-02)	2,180 4,000	3,880 5,200	3,180 4,500	1,950 3,500
Total alpha.f	(DL = 7.01E-03)	0.358	0.359	0.377	0.379
Total alpha	Calculated (Range)	0.179-0.669	0.179-0.608	0.172-0.357	0.195-0.416
$^{237}\text{Np.f}$	(DL = 3.40E-02)	< DL	< DL	< DL	< DL
$^{238}\text{Pu.f}$	(DL = 1.00E-02)	< DL	< DL	< DL	< DL
$^{239/240}\text{Pu.f}$ (325)	(DL = 3.50E-03)	0.138 0.628	0.136 0.565	0.134 0.319	0.147 0.368
$^{241}\text{Am.f}$	(DL = 3.00E-03)	0.0414	0.0431	0.0382	0.0478
$^{244}\text{Cm.f}$	(DL = Not Reported)	< DL	< DL	< DL	< DL

< DL = below detection limit

Analyte.f = fusion digestion

Analyte.a = acid digestion

Analyte.w = water digestion

Total beta calculated determined by:

1.42 (2)(^{90}Sr) + 1.51 (^{137}Cs)

Total alpha calculated determined by:

 $^{239/240}\text{Pu} + ^{241}\text{Am}$

Table 5-7. Core Composite Uranium.

Core number	U_{FL} (222-S) ($\mu\text{g/g}$)	U_{FL} (325) ($\mu\text{g/g}$)	^{238}U mass percent	^{235}U mass percent
Core 31, composite 1	2,180	4,000	99.3074	0.6755
Core 31, composite 2	3,880	5,200	99.3098	0.6761
Core 33, composite 1	3,180	4,500	99.3125	0.6761
Core 33, composite 2	1,950	3,500	99.3161	0.6717

FL = Uranium measurement by laser fluorimetry.

222-S Assay date: 4/92

325 Assay date: 9/92

Table 5-8. Plutonium Concentration and Isotopic Distribution.

Core number	Total Pu α (222-S) ($\mu\text{Ci/g}$)	Total Pu α (325) ($\mu\text{Ci/g}$)	^{238}Pu mass percent	^{239}Pu mass percent	^{240}Pu mass percent	^{241}Pu mass percent	^{242}Pu mass percent
Core 31, C1	0.138	0.628	0.005	96.7199	3.2109	0.0352	0.0151
Core 31, C2	0.136	0.565	0.0105	96.6351	3.2834	0.0496	0.0215
Core 33, C1	0.134	0.319	0.004	96.7540	3.1046	0.1071	0.0683
Core 33, C2	0.147	0.368	0.0105	96.5499	3.3436	0.0621	0.0337

222-S Assay date: 4/92

C1 = Composite 1

325 Assay date: 9/92

C2 = Composite 2

Table 5-9. Radiochemical Analyses of Grab Sample.

Analyte	Average concentration ($\mu\text{Ci/mL}$)	Average concentration ($\mu\text{Ci/g}$) ¹
^{90}Sr	0.001	0.001
^{137}Cs	0.090	0.087
$^{239/240}\text{Pu}$	6.83E-05	6.58E-05
Total Alpha	0.0024	0.0023
Total Beta	0.233	0.224

¹Density of 1.036 g/mL used for conversion.Table 5-10. Tank 241-T-111 Core 33 Radionuclide
Analyte Trending as a Function of Depth
(Acid prep on segments).

Segment	^{137}Cs ($\mu\text{Ci/g}$)	^{154}Eu ($\mu\text{Ci/g}$)	^{155}Eu ($\mu\text{Ci/g}$)	^{241}Am ($\mu\text{Ci/g}$)	^{60}Co ($\mu\text{Ci/g}$)	Total alpha ($\mu\text{Ci/g}$)
1	0.403	0.021	0.027	0.138	0.006	0.649
3	0.140	9.05E-04	0.002	0.014	0.0005	0.166
5	0.088	< DL	< DL	0.020	< DL	0.527
7	0.023	< DL	< DL	0.014	0.0005	0.350
9	0.013	< DL	< DL	0.050	< DL	0.262

5.3 TANK 241-T-111 CORE SAMPLE RHEOLOGICAL/ PHYSICAL MEASUREMENTS

Physical and rheological assays consume substantial quantities of material. Tables 5-11 and 5-12 provide a breakdown of the total amount of sample available at the outset of the analytical effort. Figure 5-1 shows the location where the core samples were taken and the waste depth. Measurements of physical characteristics such as shear strength, viscosity, particle size, and settling properties were taken. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and final waste disposal systems. Rheological assays were performed on samples from core 31, segments 2, 4, and 8. Particle size measurements were done on each segment of core 31. The data from segment 4 is not considered valid for these assays because it had dried before the measurements were taken. The drying process irreversibly changed the physical properties of the sample under investigation, and thus the sample is not considered representative. Therefore, the results from most of these assays will not be presented. However, in some cases it is useful to compare and contrast the results from the "representative" samples with the samples that dried.

Table 5-11. Tank 241-T-111 Core Sample Description Summary.

Core number	Segment number	Solid sample mass (g)	Liquid sample mass (g)	Approximate solid sample volume (mL)	Liquid sample volume (mL)	Approximate solid sample length
31	1	18.74	45.26	Not Recorded	~ 50	Not Resolved
31	2	178.68	4.12	150	NA	15 in. (38 cm)
31	3	162.2	NA	177	NA	18 in. (46 cm)
31	4	153.47	NA	148	NA	15 in. (38 cm)
31	5	190.94	NA	187	NA	19 in. (48 cm)
31	6	0	NA	0	NA	No Sample
31	7	186.44	NA	177	NA	18 in. (46 cm)
31	8	186.44	NA	187	NA	19 in. (48 cm)
31	9	203.08	NA	187	NA	19 in. (48 cm)

Figure 5-1. Current Condition of Tank 241-T-111.

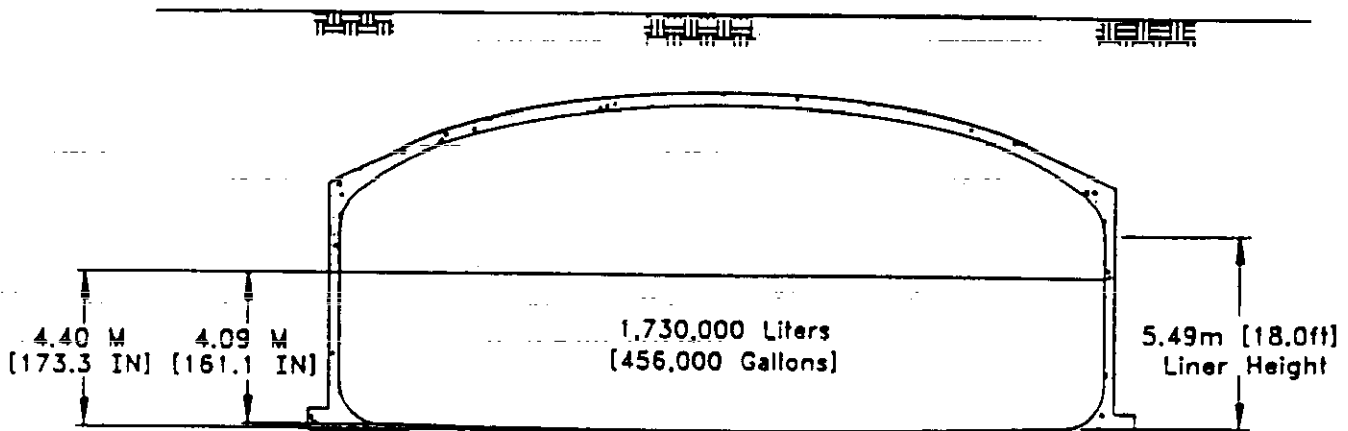
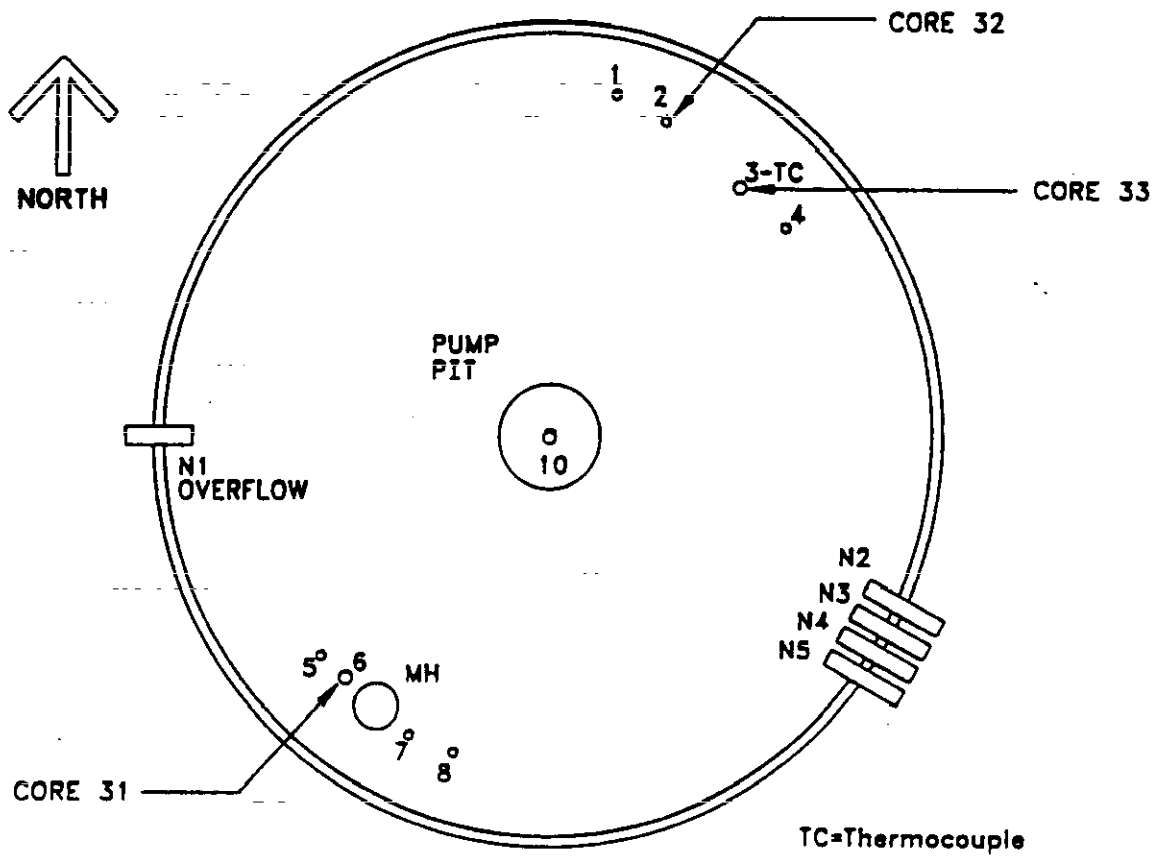


Table 5-12. Tank 241-T-111 Core Sample Description Summary.

Core number	Segment number	Solid sample mass (g)	Liquid sample mass (g)	Approximate solid sample volume (mL)	Liquid sample volume (mL)	Approximate solid sample length
33	1	159.16	0	137	NA	14 in. (36 cm)
33	2	209.59	0	187	NA	19 in. (48 cm)
33	3	167.9	0	163	NA	16 in. (41 cm)
33	4	182.05	0	167	NA	16 in. (41 cm)
33	5	174.3	0	167	NA	17 in. (43 cm)
33	6	217.37	0	187	NA	19 in. (48 cm)
33	7	196.91	0	187	NA	19 in. (48 cm)
33	8	199.8	0	187	NA	19 in. (48 cm)
33	9	191.01	0	187	NA	19 in. (48 cm)

5.3.1 Shear Strength

The shear strength of the waste from tank 241-T-111 was measured on the unhomogenized segment samples from core 31 (segments 2, 4, and 8). The shear strength measurements were made at ambient temperatures using a shear vane connected to a viscometer and rotated at 0.3 rpm. Shear strength (τ_s) is a semiquantitative measurement of the force required to move the sample. Because shear strength is dependent on sample handling, the measurement was taken without any sample homogenization. Small aliquots from the segments 2, 4, and 8 from core 31 were taken and assayed. The aliquots were transferred to a sample jar and allowed to settle for several weeks so that they could recover from the disturbance of sampling and extrusion. The extended delay between sample and analysis was permitted because it is believed that the longer the sample sits undisturbed, the closer it will resemble its original condition; therefore, the shear measurement is likely to be more representative. The shear stress (S_τ) of the sample was recorded as a function of time and the shear strength was calculated using Equation 1.

$$\tau_s = \frac{[\% \tau / 100] * S_\tau * 4.9E+05}{\frac{\pi * H_v * D_v^2}{2} + \frac{\pi * D_v^3}{6}} \quad (1)$$

where:

$\% \tau / 100$ = The ratio of the total torque to the maximum torque of the viscometer head, measured as a percentage of the full scale on the plot of the shear stress versus time diagram (dimensionless)

S_r = Instrument reading proportional to the torque (dimensionless)

$4.9E+05$ = maximum torque of the viscometer head (dyne•cm)

H_v = shear vane height (0.635 cm)

D_v = shear vane diameter (0.635 cm)

The shear strength for segments 2 and 8 were found to range $5,000 \pm 2,300$ dynes/cm². Segment 4 is not reported because of sample drying. Although relatively low, the shear stress of the material exceeded the baseline value for the measurement system (200 dynes/cm²). Therefore, the values are considered to be valid and representative. Some additional drying of the sample may have occurred during the settling time, causing the shear stress to be higher than expected.

5.3.2 Shear Stress and Viscosity as Functions of Shear Rate

Shear stress measurements, as functions of shear rate, were performed on the as-received, 1 to 1 and 3 to 1 water to sample dilution of the sample at ambient hot-cell temperatures [ranging from 27 to 34 °C (81 to 93 °F)] and at 95 °C (203 °F). Drying the as-received sample at 95 °C posed difficulties in measurement; therefore no results of the rheograms for the samples run under those conditions are presented. In addition, the results from segment 4 are not presented because the drying that occurred compromised the sample properties, as discussed previously.

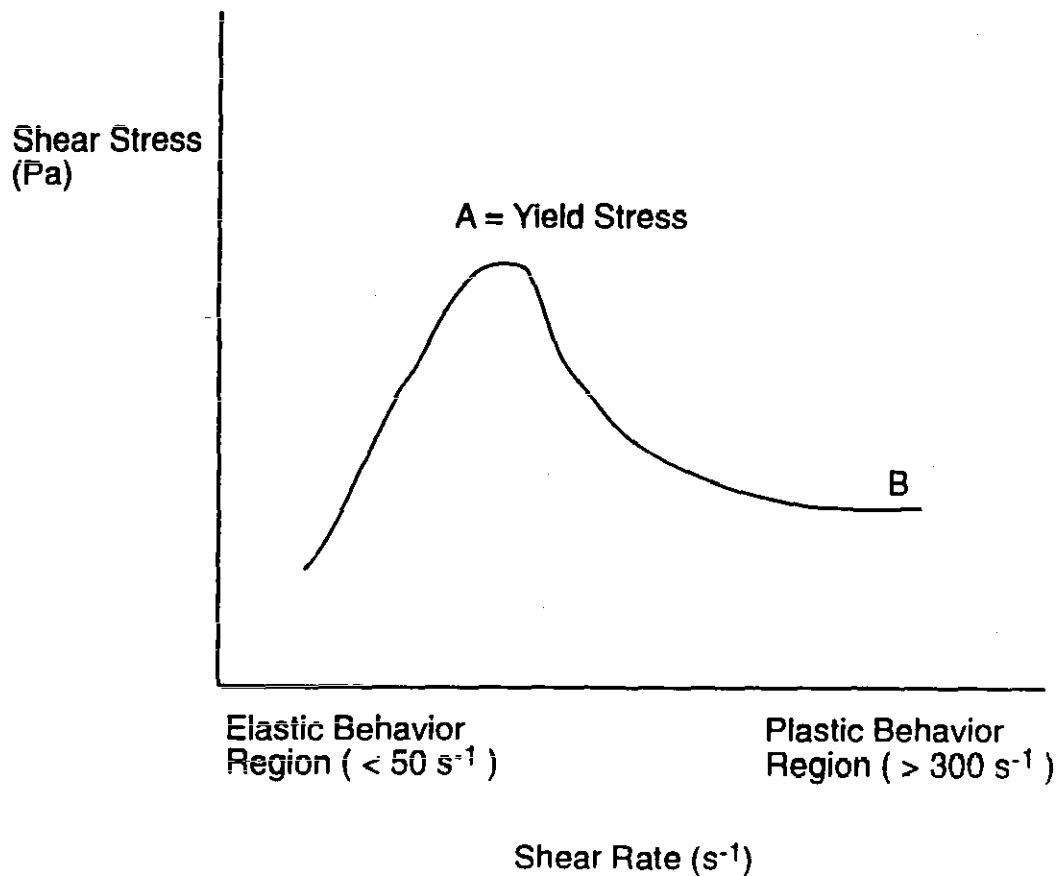
A rheogram for a material with a yield stress has two sections. The first section is a straight line beginning at the origin and climbing up the ordinate. This portion of the rheogram records the material as it acts like a solid or gel. When sufficient force is applied to the material to make it yield, the rheogram breaks sharply to the right, recording the material's behavior as a fluid. The point on the rheogram at which the sample's behavior transfers from a solid or gel to a fluid is the yield point or yield stress. This minimum shear stress must be exceeded to initiate fluid behavior. The tank wastes demonstrate both elastic and plastic behavior, depending on the amount of shear acting on them. The samples are elastic under low shear conditions (less than 50 s⁻¹), and plastic under high shear conditions (greater than 300 s⁻¹).

Viscosity measurements as a function of shear rate for the 1 to 1 diluted samples had viscosities near the limits of detection of the system (2 cP) for over the broad range of shear

rates; however, some qualitative and quantitative information was obtained from the measurement trials. Even though the measurements were at the limits of detection, the qualitative and quantitative behavior was consistent and reproducible. Viscosity was observed to increase slightly, then decrease with increasing shear rates. The 1 to 1 dilution of the segment 2 and segment 8 samples exhibited tendencies toward yield-pseudoplastic behavior. In fact, the general behavior exhibited by the wastes is best described by a yield-pseudoplastic model, however the system was not modeled and empirical model parameters were not determined because the system was at the detection limits. No other measurements of the viscosity as a function of shear rate were made on the 1 to 1 dilution samples at 95 °C (203 °F) or the 3 to 1 dilution samples.

Figures 5-2 to 5-6 are general illustrations of the rheograms. They are not to scale and do not fully capture all of the nuances and detail that is contained in each measurement trial. However, when coupled with the description underneath each diagram, much insight can be obtained about the flow properties of the waste. If more detailed rheological information is required, the data package should be consulted. Note that in the figures, Point A is where the sample begins to register movement. Point B represents the behavior of the sample at the maximum shear rate of the viscometer.

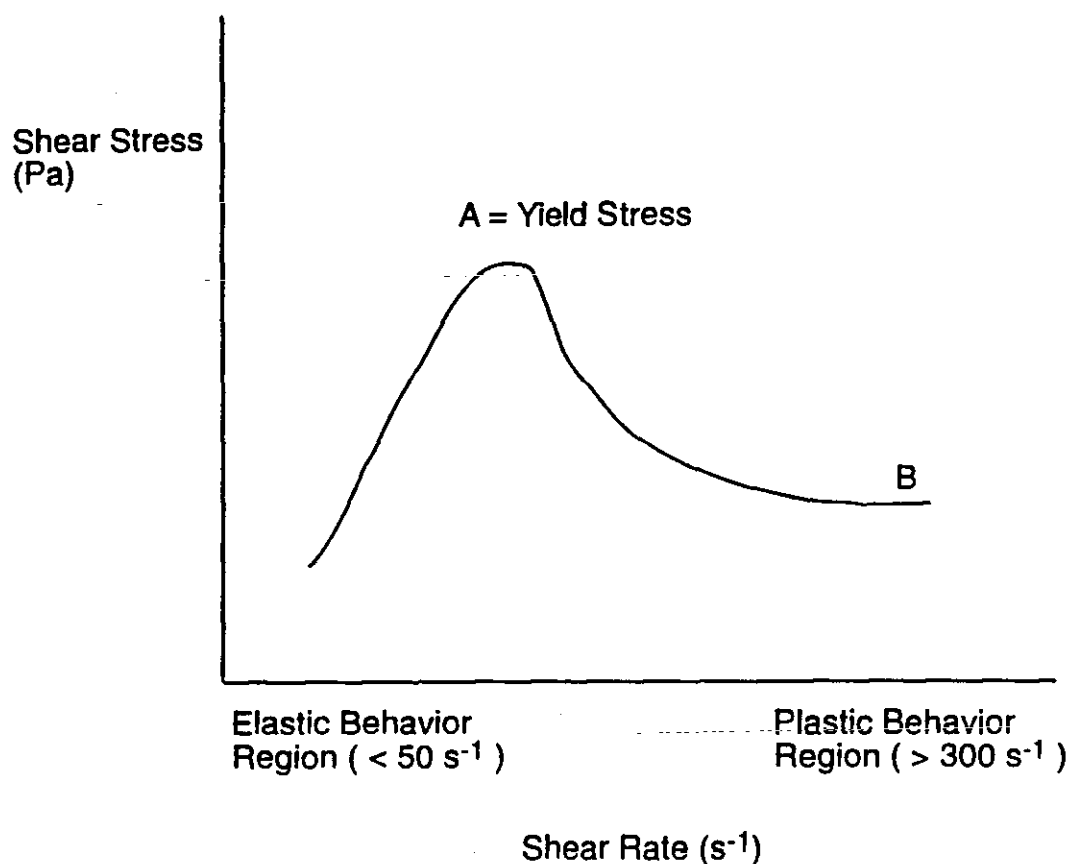
Figure 5-2. Shear Stress as a Function of Shear Rate: Direct Sample.



Shear Stress as a Function of Shear Rate: Direct Sample.

Sample: core 31	Sample number	Temperature (°C)	Point A shear stress range (Pa)	Qualitative behavior of rheogram	Point B shear stress (Pa)
Segment 2	80701	34	88 - 220	Wide variation at low shear, converging to a single value at high shear.	165
Segment 2	80703	33	200 - 680	Same	70
Segment 8	123201	33	36 - 108	Same	77
Segment 8	123202	33	0 - 108	Same	50

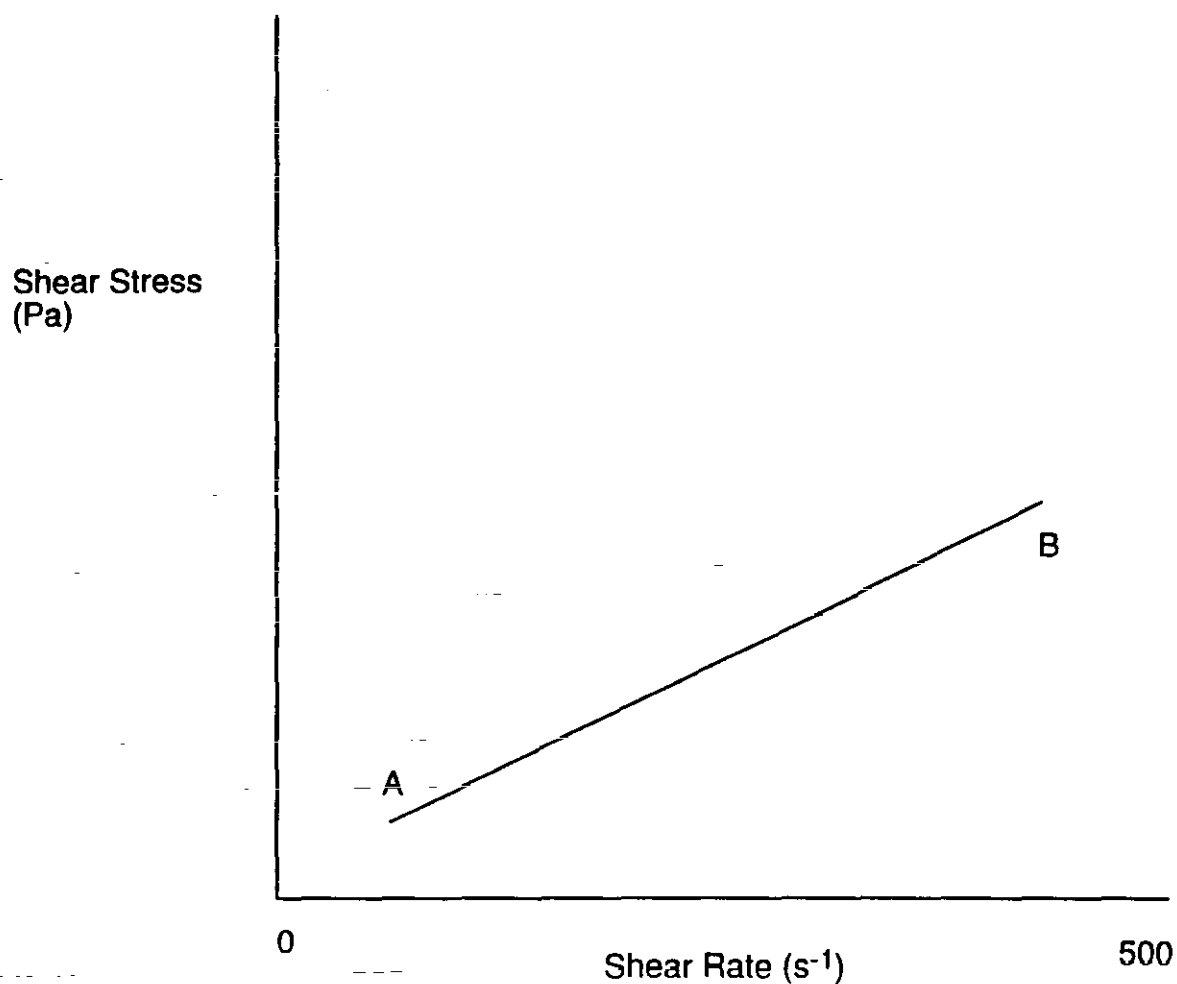
Figure 5-3. Shear Stress as a Function of Shear Rate: 1 to 1 Dilution, Water to Sample.



Shear Stress as a Function of Shear Rate: 1 to 1 Dilution, Water to Sample.

Sample: core 31	Sample number	Temperature (°C)	Point A shear stress range (Pa)	Qualitative behavior of rheogram	Point B shear stress (Pa)
Segment 2	1	27	0.6 - 4.2	Wide variation at low shear, converging to a single value at high shear.	2.4
Segment 2	2	27	1.2	Linear	2.8
Segment 2	3	95	1.0 - 7.0	Wide variation at low shear, converging to a single value at high shear.	2.0
Segment 2	4	95	0.7 - 1.1	Linear	1.2 - 1.4
Segment 2	5	95	1.0 - 2.4	Linear	2.2

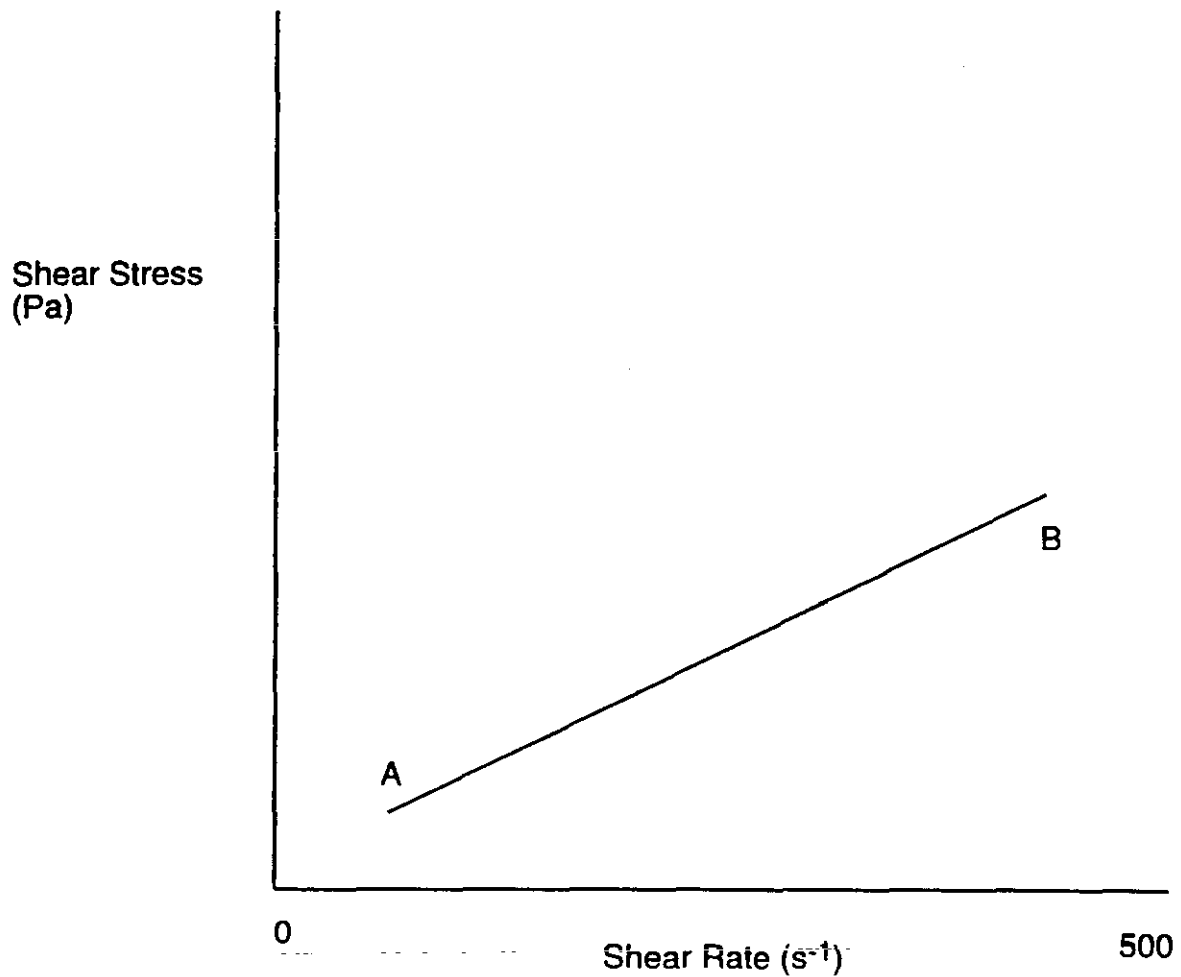
Figure 5-4. Shear Stress as a Function of Shear Rate: 1 to 1 Dilution, Water to Sample.



Shear Stress as a Function of Shear Rate: 1 to 1 Dilution, Water to Sample

Sample: core 31	Sample number	Temperature (°C)	Point A shear stress range (Pa)	Qualitative behavior of rheogram	Point B shear stress (Pa)
Segment 8	1	27	0.4 - 0.6	Linear	2.8
Segment 8	2	27	0.6	Linear	2.8
Segment 8	3	95	0.6	Linear	2.0
Segment 8	4	95	2.0 - 5.0	Erratic, non-linear	2.0
Segment 8	5	95	0.2	Linear	0.7 - 0.9

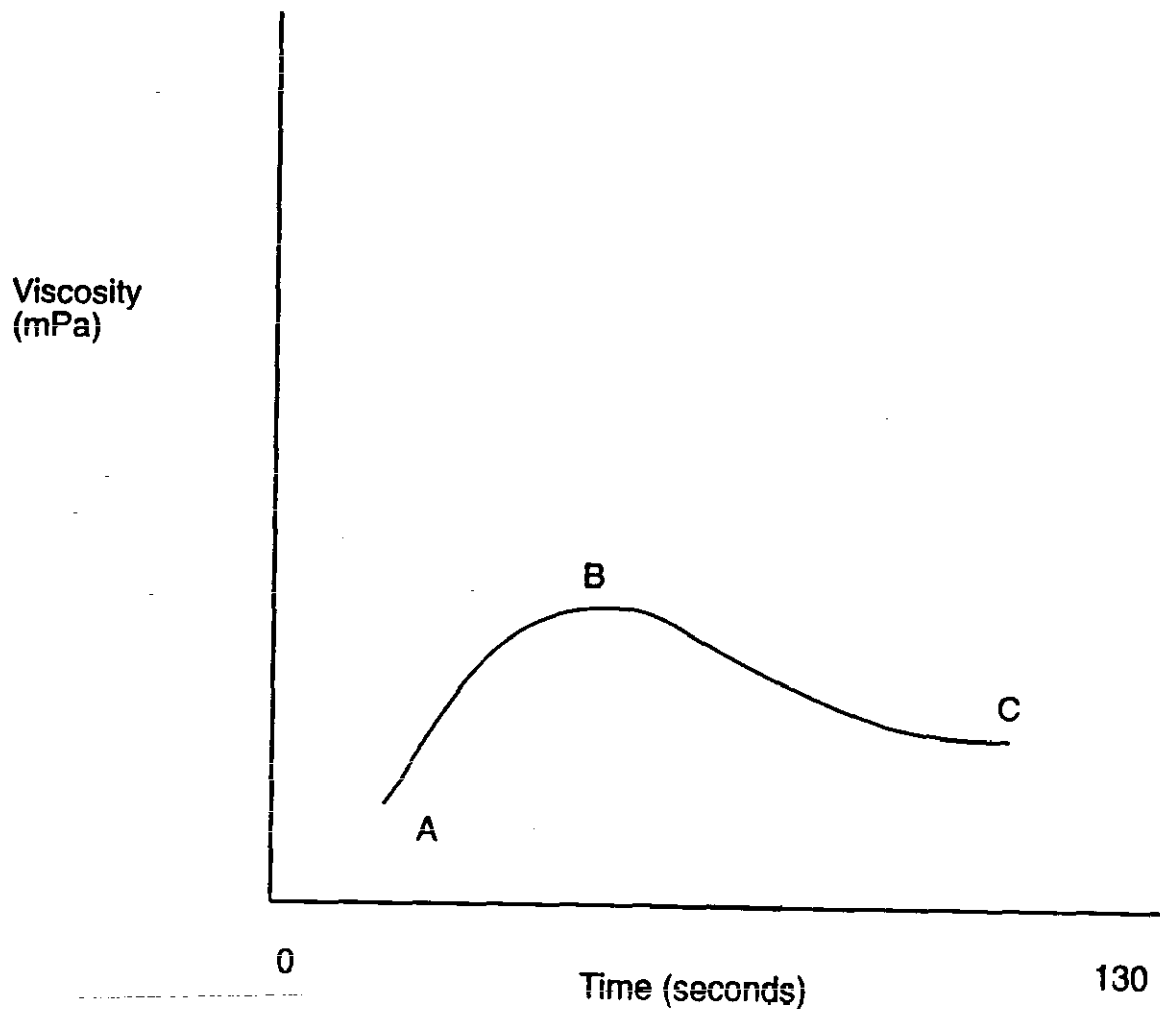
Figure 5-5. Shear Stress as a Function of Shear Rate: 3 to 1 Dilution, Water to Sample.



Shear Stress as a Function of Shear Rate: 3 to 1 Dilution, Water to Sample.

Sample: core 31	Sample number	Temperature (°C)	Point A shear stress range (Pa)	Qualitative behavior of rheogram	Point B shear stress (Pa)
Segment 2	1	27	0.05 - 0.25	Linear	0.75 - 1.1
Segment 2	2	27	0.2 - 0.35	Linear	1.2
Segment 2	3	95	0.2	Linear	0.8
Segment 2	4	95	0.3	Linear	1.0
Segment 8	3	95	Not defined	Erratic, non-linear	0.4
Segment 8	4	95	0.3	Linear	1.0

Figure 5-6. Viscosity as a Function of Time: 1 to 1 Dilution, Water to Sample.



Viscosity as a Function of Shear Rate: 1 to 1 Dilution, Water to Sample

Sample: core 31	Sample number	Temperature (°C)	Point A viscosity (mPa)	Qualitative description of rheogram	Point B viscosity (mPa)	Point C viscosity (mPa)
Segment 2	1	30	0.65	Rises, levels off, then gradually declines	1.8 - 2.0	1.5 - 1.7
Segment 2	2	30	0.56	Slightly sinusoidal	0.6	0.6
Segment 2	3	30	0.80	Rises, levels off, then gradually declines	1.0	0.9
Segment 8	3	30	0.75	Flattened exponential growth and decay curve	1.0	0.75
Segment 8	4	30	0.85	Flattened exponential growth and decay curve	0.95	0.9

5.3.3 Particle Size Measurement

Particle size is analyzed by placing a small amount of sample in a dispersant, which is the liquid used to disperse and suspend the particles from the solid sample. Samples from each segment of core 31 were prepared and assayed. The prepared sample was placed in a particle size analyzer. The apparatus measures particle size by passing a thin beam of laser light through the dispersant. The diameter of a particle in the dispersant can be determined by the amount of light that it blocks as it passes through the beam. The dimension measured by this method is the value across the short diameter of the particle. This means that if a particle is oblong, the machine estimates the shortest length across the particle. The term "diameter" throughout this text will be used to describe any linear profile of any shape.

An important consideration involving the analysis of particle size is the dispersant used. The primary concern involved with the dispersant is that it may dissolve the particle. Any particles existing in the tank that are soluble in the dispersant will dissolve or decrease in size during the analysis. Depending on the dispersant, the particle size analysis may not represent the true particle size distribution in the tank. In the case of tank 241-T-111, water was used as the dispersing medium. If a true particle size distribution is required, the mother liquor, or drainable liquid of the tank should be used if possible because the tank particulates are already in equilibrium with the tank's mother liquor. The insolubility of the waste matrix suggests that the particle size data acquired should be acceptable.

The mean particle size in the number distribution runs in a narrow range from 0.93 to 1.23 microns in diameter for tank 241-T-111 waste samples. Table 5-13 presents the summary results of the measurements.

Table 5-13. Core 31 Particle Size Distribution by Number.

Segment	Mean (μm)	Standard deviation	Median (μm)
1	1.23	0.89	0.94
2	1.13	0.80	0.88
3	1.17	1.00	0.91
4	0.93	0.60	0.80
5	0.95	0.63	0.81
6	-	-	-
7	0.97	0.60	0.83
8	1.02	0.85	0.82
9	1.02	0.83	0.83

Table 5-14 presents the summary results of the volume distribution measurements. Assuming that the density of the solid material within the tank is constant, the volume distribution is also the best estimation of the mass particle size distribution of the tank.

Table 5-14. Core 31 Particle Size Distribution by Volume.

Segment	Mean (μm)	Standard deviation	Median (μm)
1	28.56	35.92	5.81
2	14.91	20.76	4.79
3	64.99	46.19	58.69
4	24.87	34.15	5.63
5	37.87	47.91	12.31
6	-	-	-
7	7.95	11.88	4.02
8	24.72	28.18	10.02
9	59.69	49.04	58.97

The number density graph is plotted over the acquisition range of the device (from 0.5 to 150 microns). The numbers of particles in each size range, shown as a percentage of the whole, are graphed against their respective size ranges to form a distribution curve. The figures show that the modes for particle size range between the origin and 2.0 microns. In

fact, over 80 percent of the measured particles fit within this narrow band. As with the number distribution, the volume distribution is represented by a probability volume density graph. The average particle size in the volume distribution is considerably larger than in the number distribution. The particle size in the volume distribution ranges over the full scale of the device, 0.0 microns to 150 microns in diameter, between the eight segments (core 31 segment 6 was empty, but the nomenclature for the other samples held). The analyzer calculates particle volume as the cube of the diameter.

In core 31, approximately 70 percent of particle measurements for segments 1, 2, 4, 5, 7, and 8 fall into the range between 0.0 and 24.0 microns, and tend to be dispersed towards the smaller particle sizes. Segments 3 and 9 were exceptions to this rule. They had a distribution over the broad spectrum of particle sizes, with the particles generally much larger and more widely scattered over the 0.0 to 150.0 micron range and a slight tendency towards the smaller end of the scale. In segment 3, 23 percent of the particles were less than 24 microns. In segment 9, 40 percent of the particles were less than 24 microns.

The disparity between the segment measurements possibly indicates a difference in waste type, or perhaps a transitional layer between two waste types that individually are physically similar, but when commingled, may precipitate larger particulate materials. In segments 1, 2, 4, 5, 7, and 8, over 60 percent of the particles in the sample have a diameter of less than 24.0 microns. In segments 3 and 9, over 50 percent of the particles have a diameter of greater than 24.0 microns. In the retrieval and subsequent treatment of the tank wastes, it may be desirable to design pumping or filtration systems for the tank particulate. Therefore, the volume distribution of the particles should not be neglected (i.e., particles with diameters of over 20 microns should be considered in these designs). In addition, variation in particle size distribution is believed to have an impact on analytical precision, especially with small sample sizes, and thus, should be considered when evaluating analytical results. Plots of the probability number and volume-density for each core are presented as Figures 5-7a to 5-14b.

5.3.4 Settling Behavior of As-Received and Diluted Samples

This section analyzes the settling behavior and physical properties of the grab samples and the as-received 1 to 1 and 3 to 1 water to sample dilutions. The physical properties reported here include settling rates and volume percent for settled solids and weight percent and volume percent for centrifuged solids. The experimental procedures used to take these measurements were reported previously (McKinney et. al 1993). The physical properties of the grab samples are reported in Table 5-15. The properties for core 31 samples are summarized in Table 5-16.

Figure 5-7a. Core 31, Segment 1, Particle Size: Number Density.

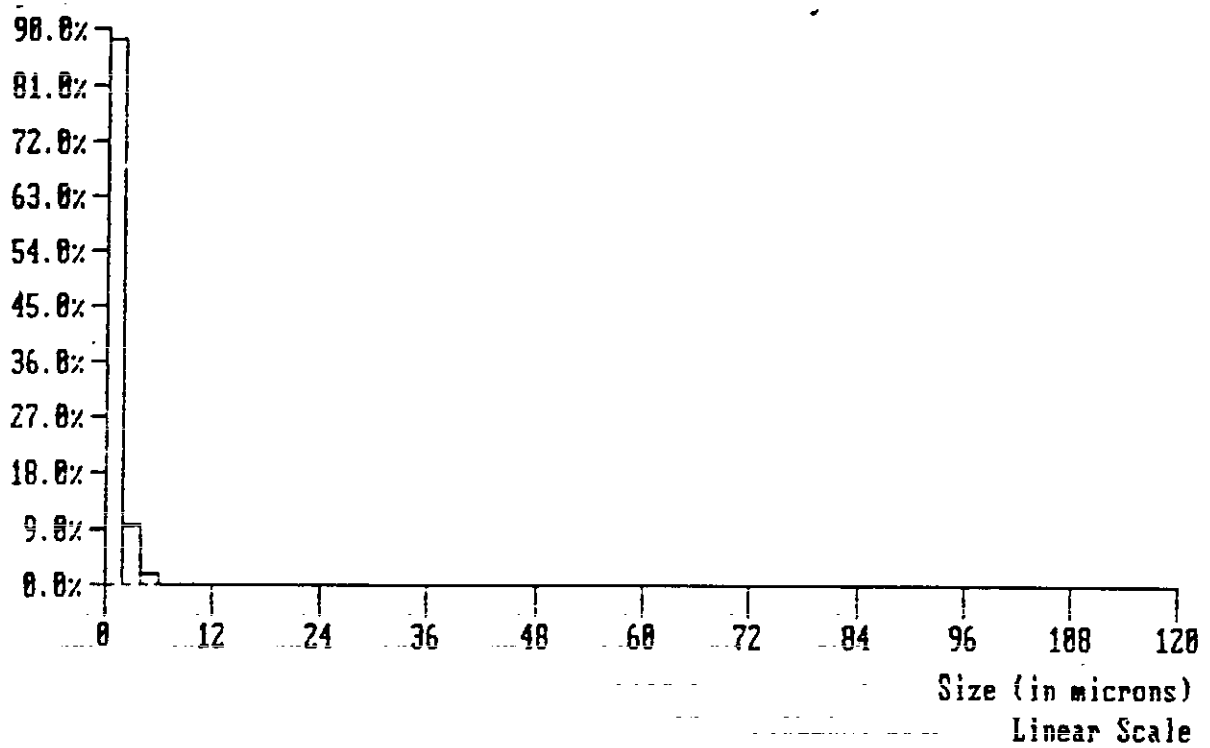


Figure 5-7b. Core 31, Segment 1, Particle Size: Volume Density.

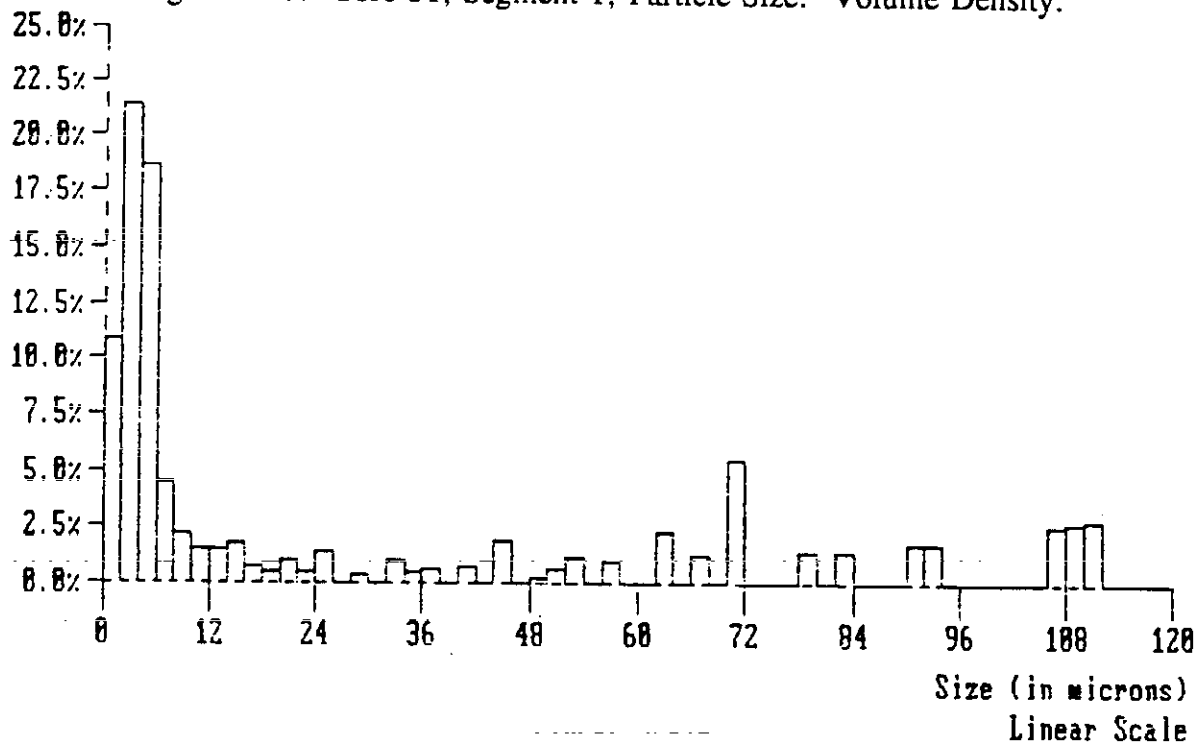


Figure 5-8a. Core 31, Segment 2, Particle Size: Number Density.

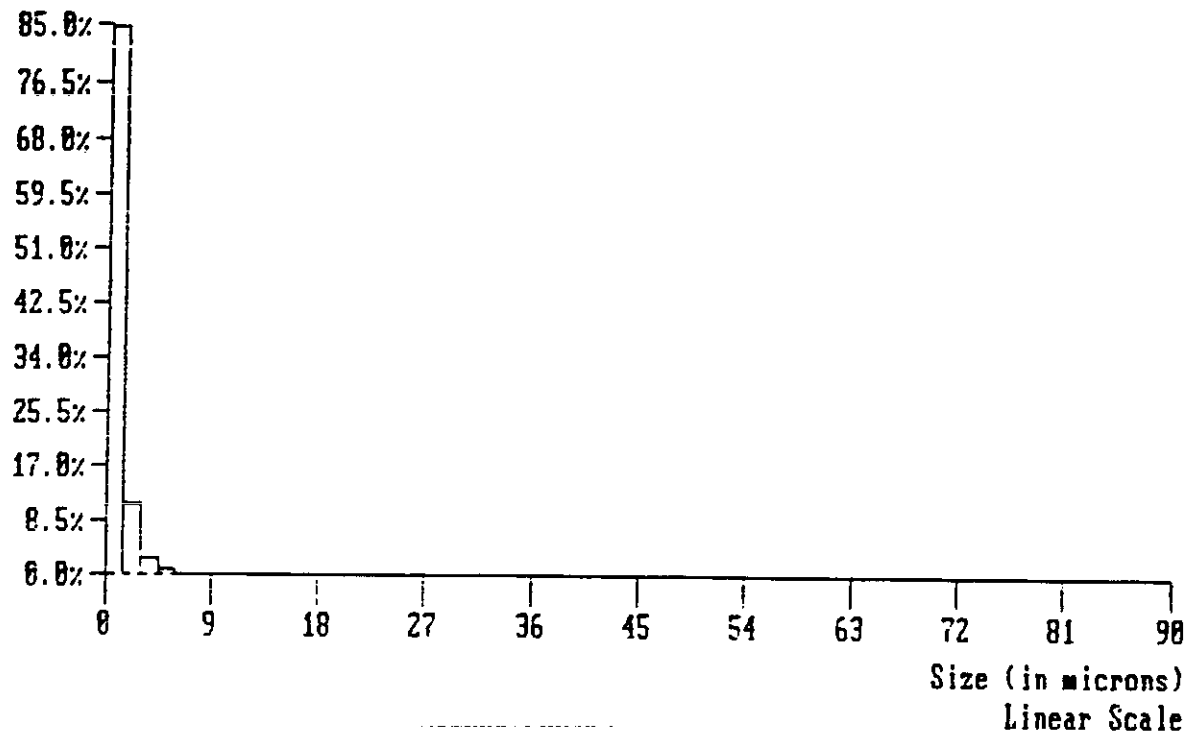


Figure 5-8b. Core 31, Segment 2, Particle Size: Volume Density.

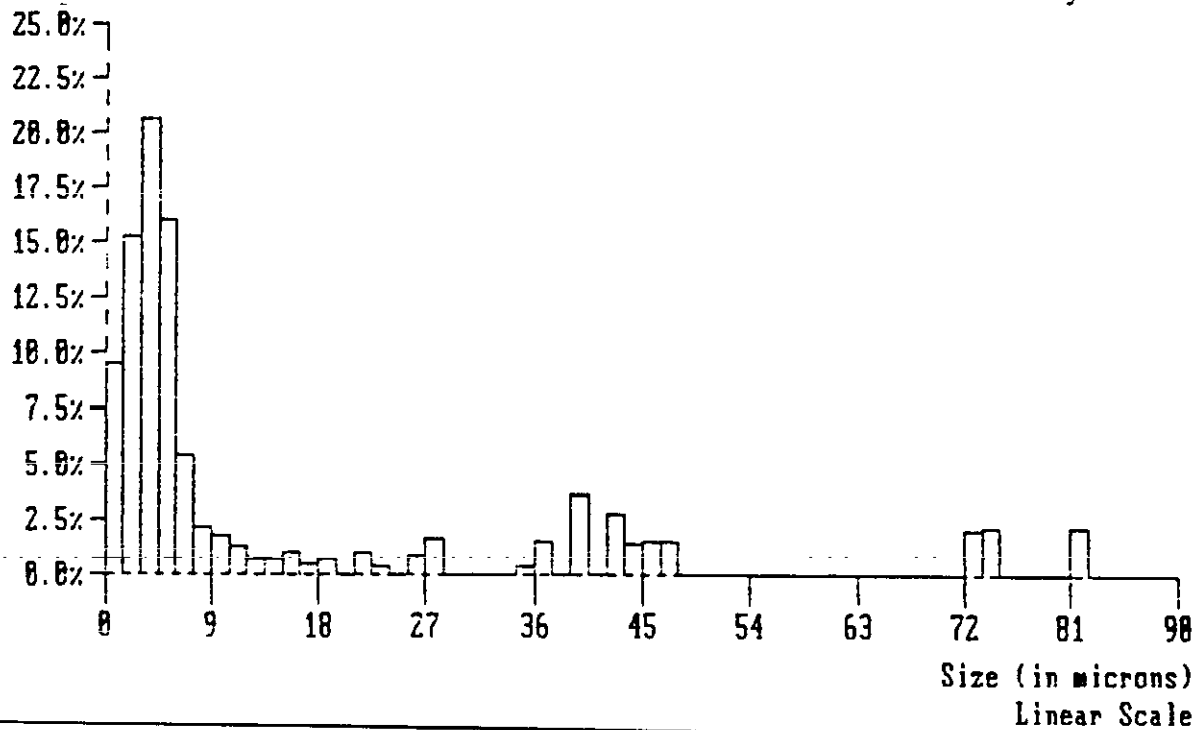


Figure 5-9a. Core 31, Segment 3, Particle Size: Number Density.

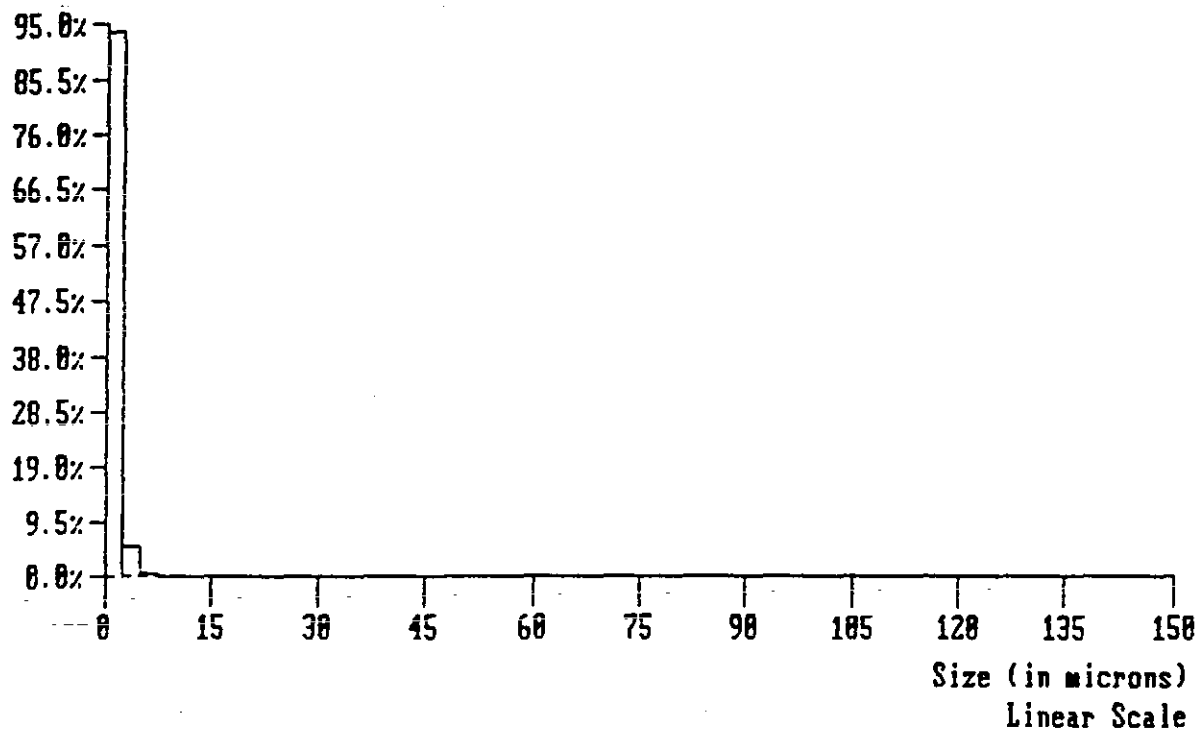


Figure 5-9b. Core 31, Segment 3, Particle Size: Volume Density.

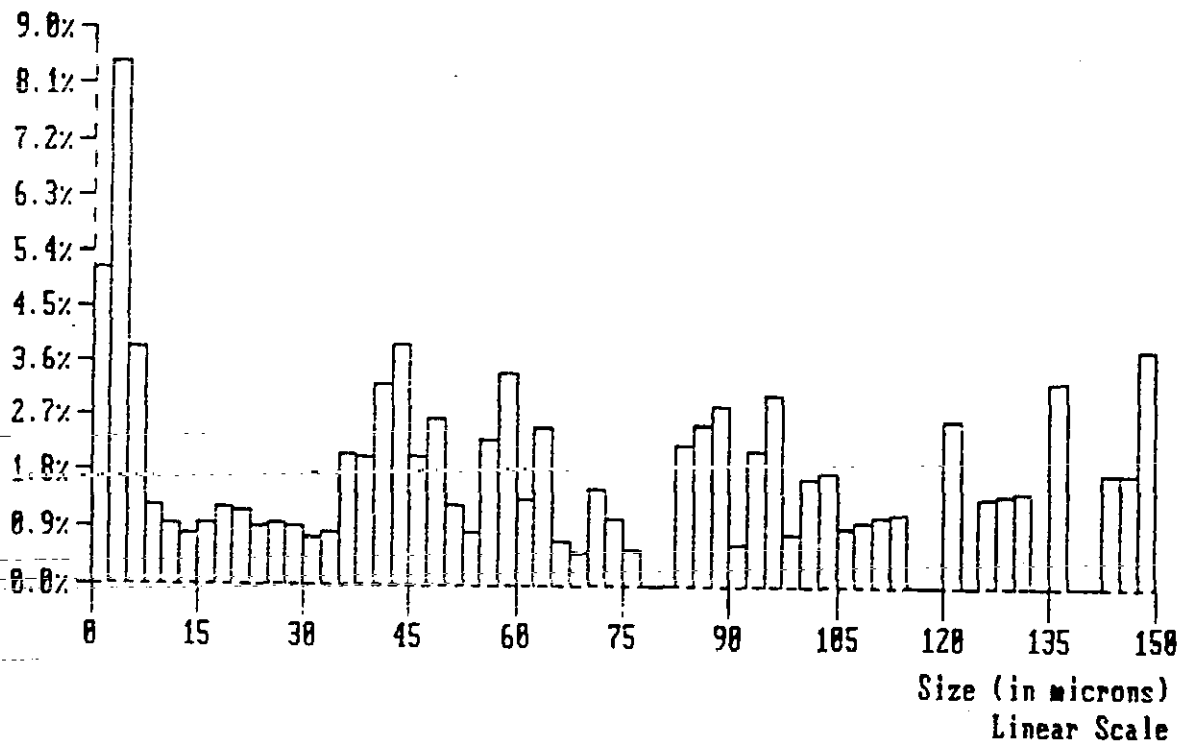


Figure 5-10a. Core 31, Segment 4, Particle Size: Number Density.

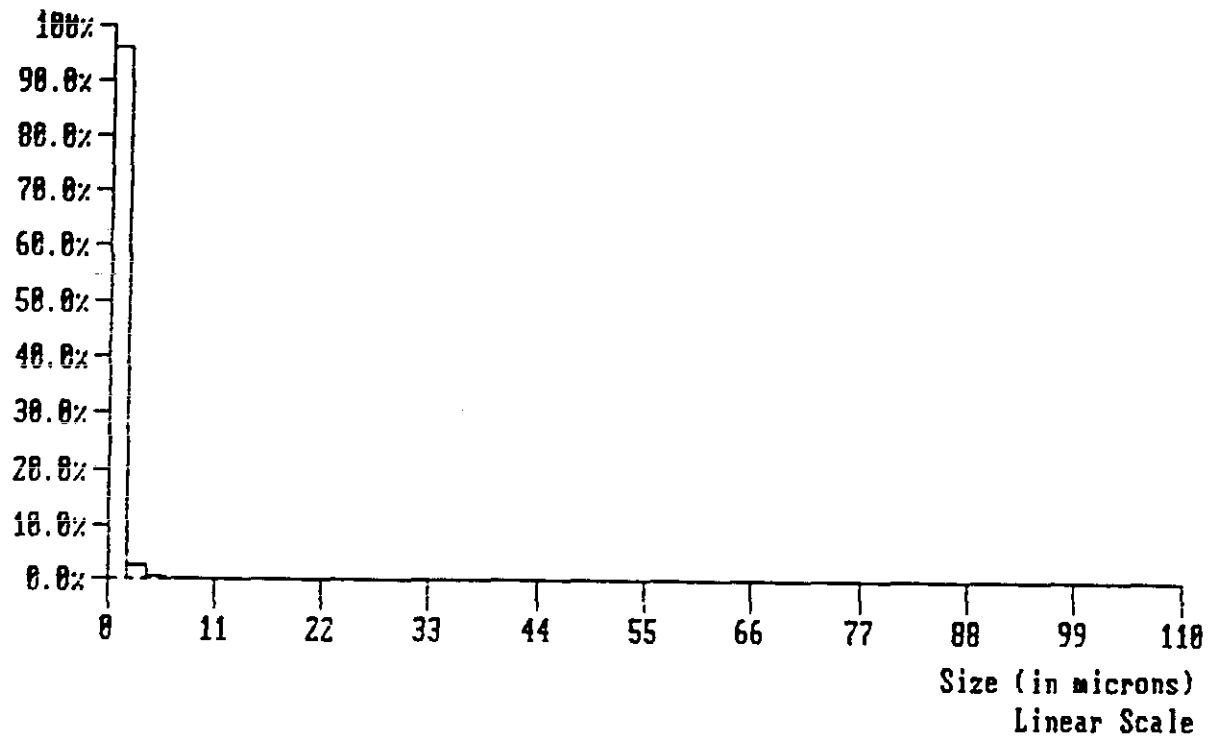


Figure 5-10b. Core 31, Segment 4, Particle Size: Volume Density.

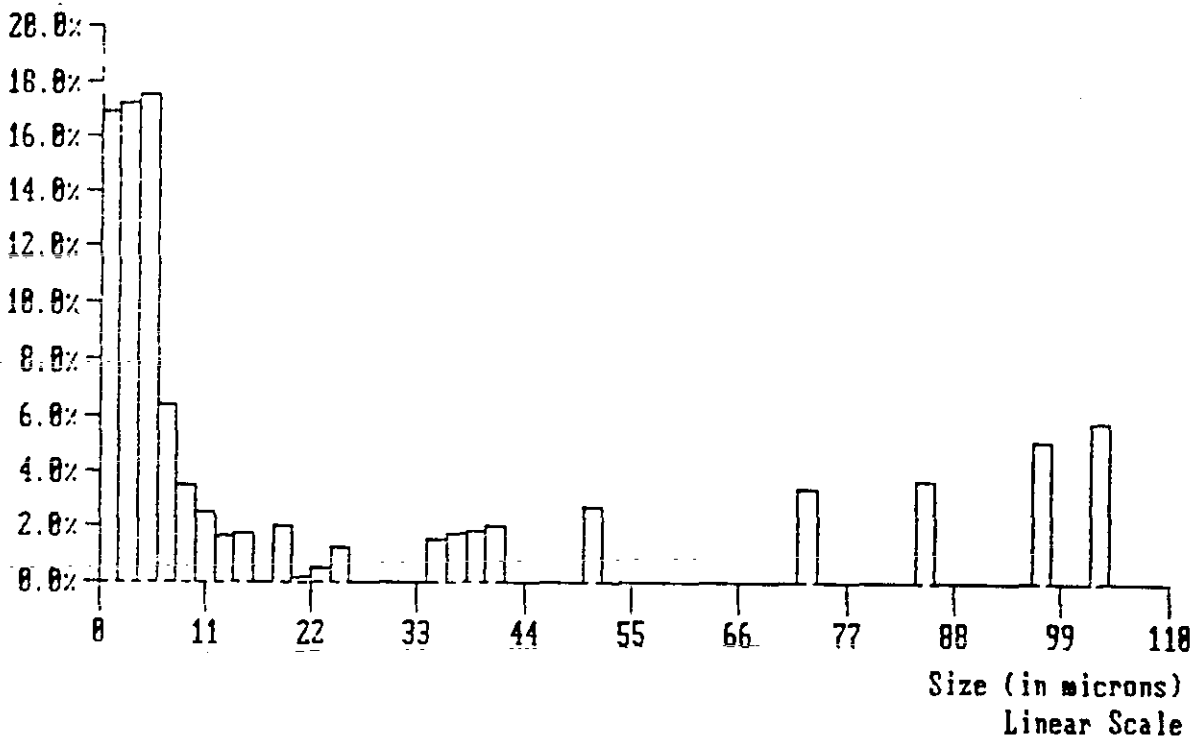


Figure 5-11a. Core 31, Segment 5, Particle Size: Number Density.

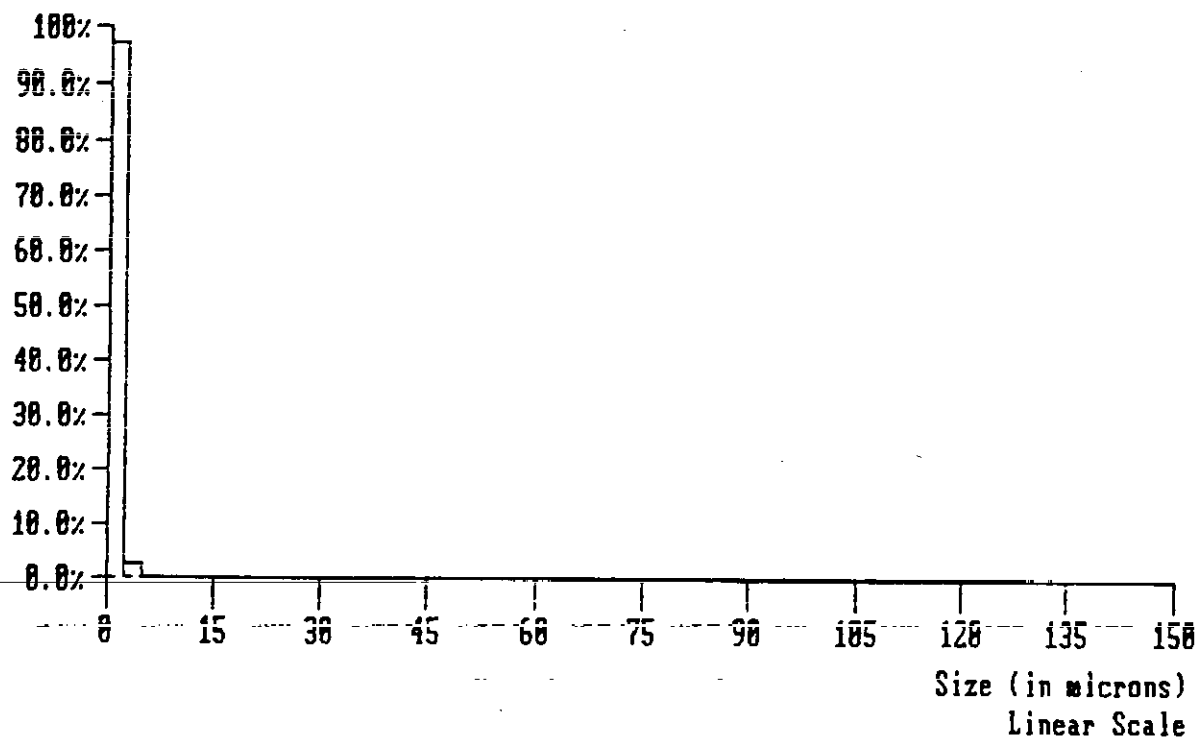


Figure 5-11b. Core 31, Segment 5, Particle Size: Volume Density.

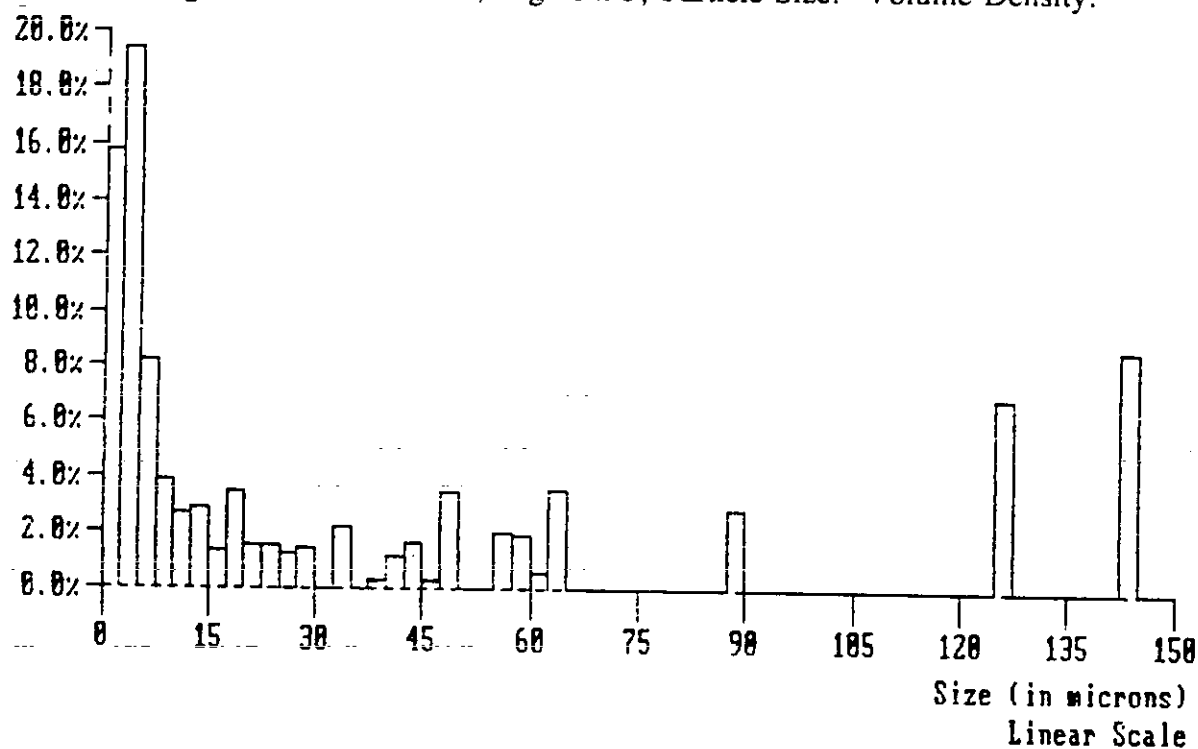


Figure 5-12a. Core 31, Segment 7, Particle Size: Number Density.

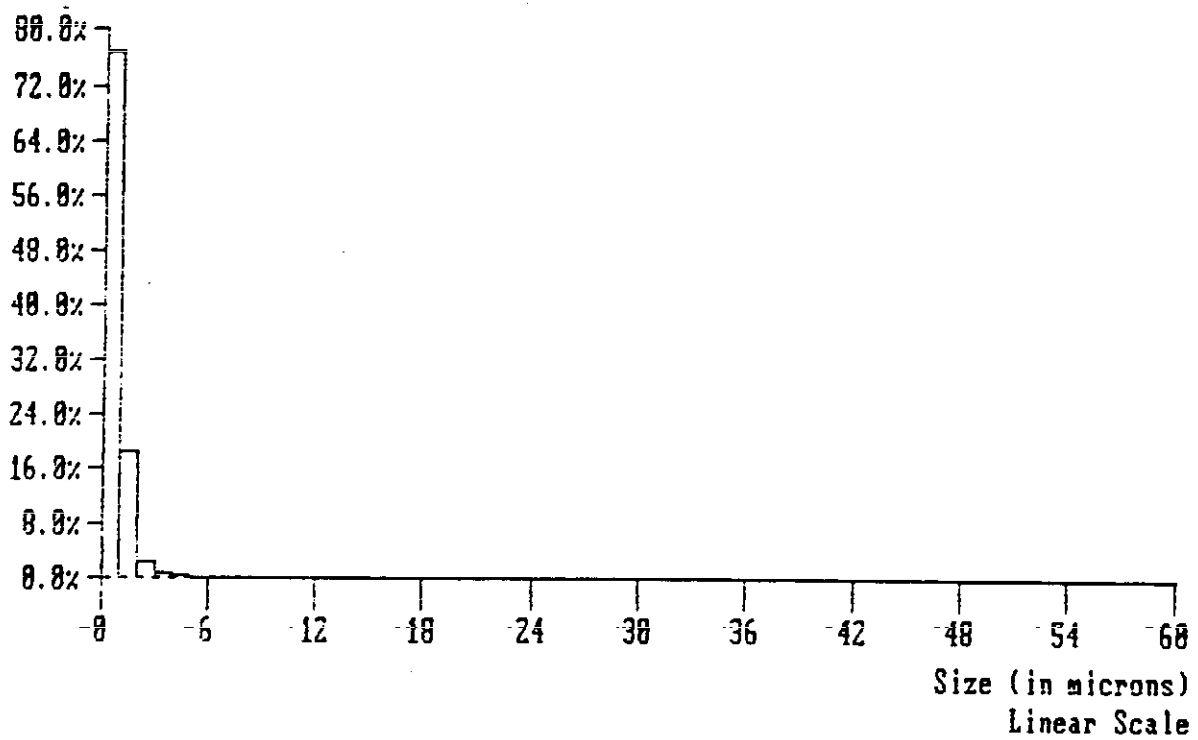


Figure 5-12b. Core 31, Segment 7, Particle Size: Volume Density.

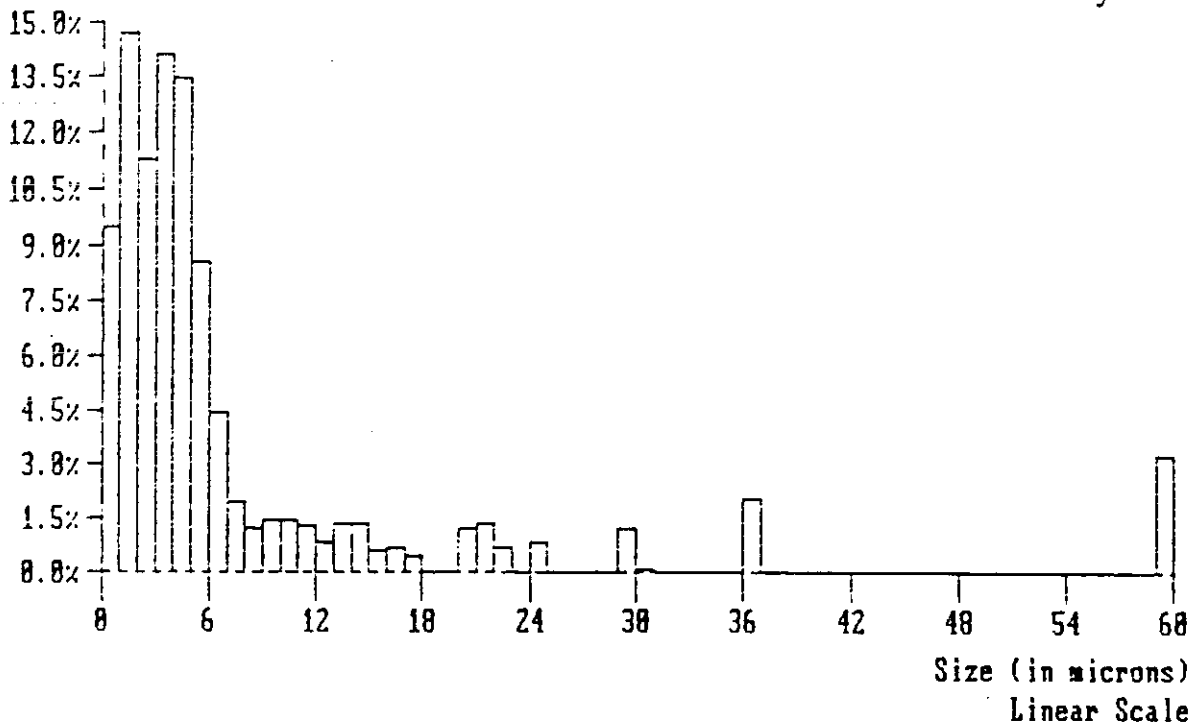


Figure 5-13a. Core 31, Segment 8, Particle Size: Number Density.

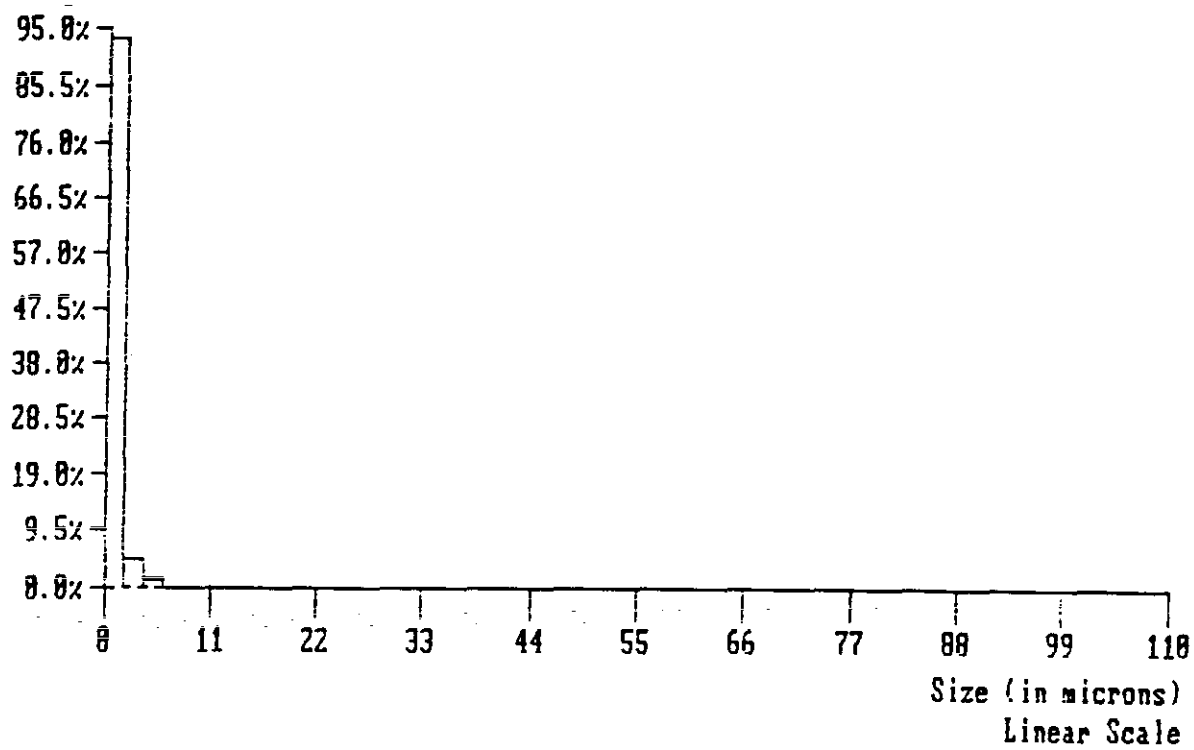


Figure 5-13b. Core 31, Segment 8, Particle Size: Volume Density.

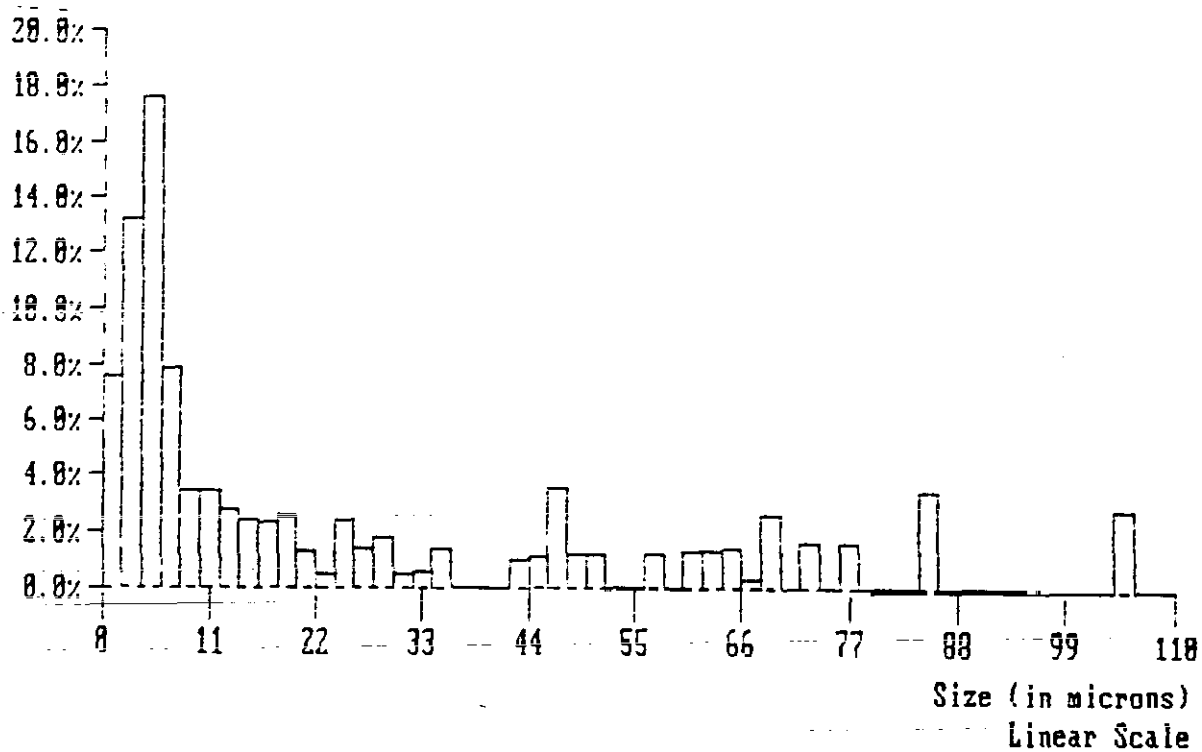


Figure 5-14a. Core 31, Segment 9, Particle Size: Number Density.

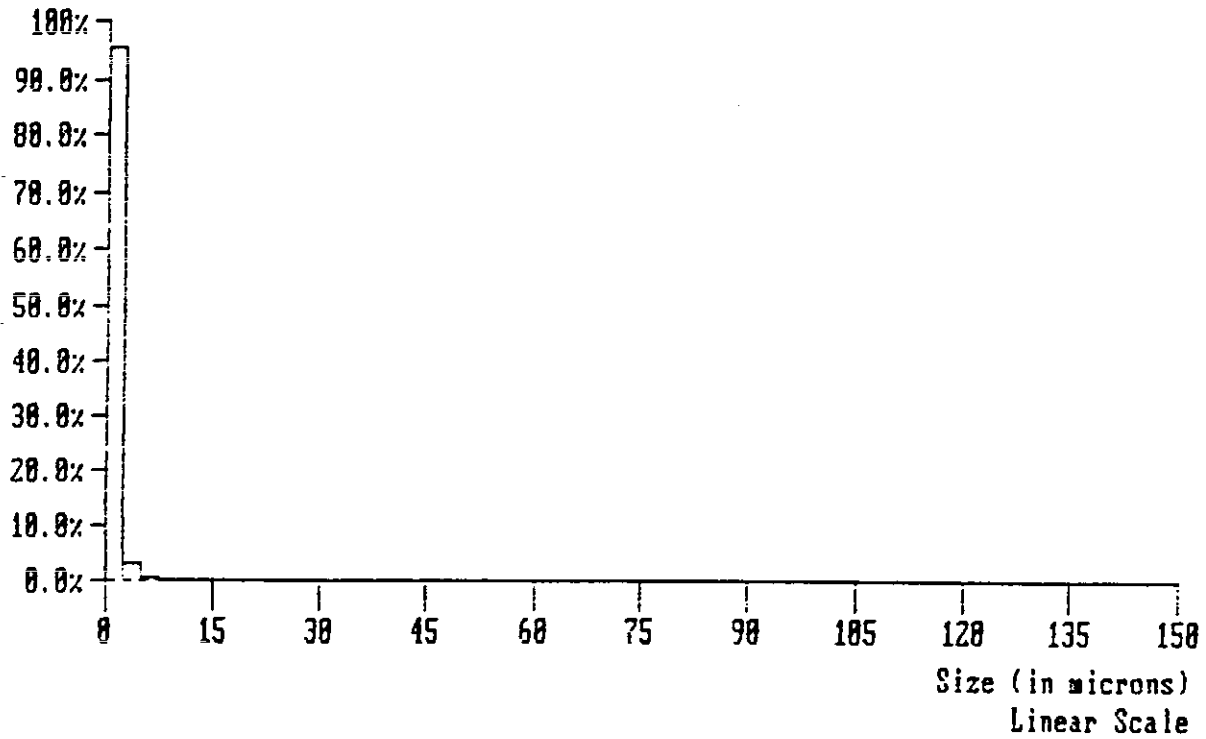


Figure 5-14b. Core 31, Segment 9, Particle Size: Volume Density.

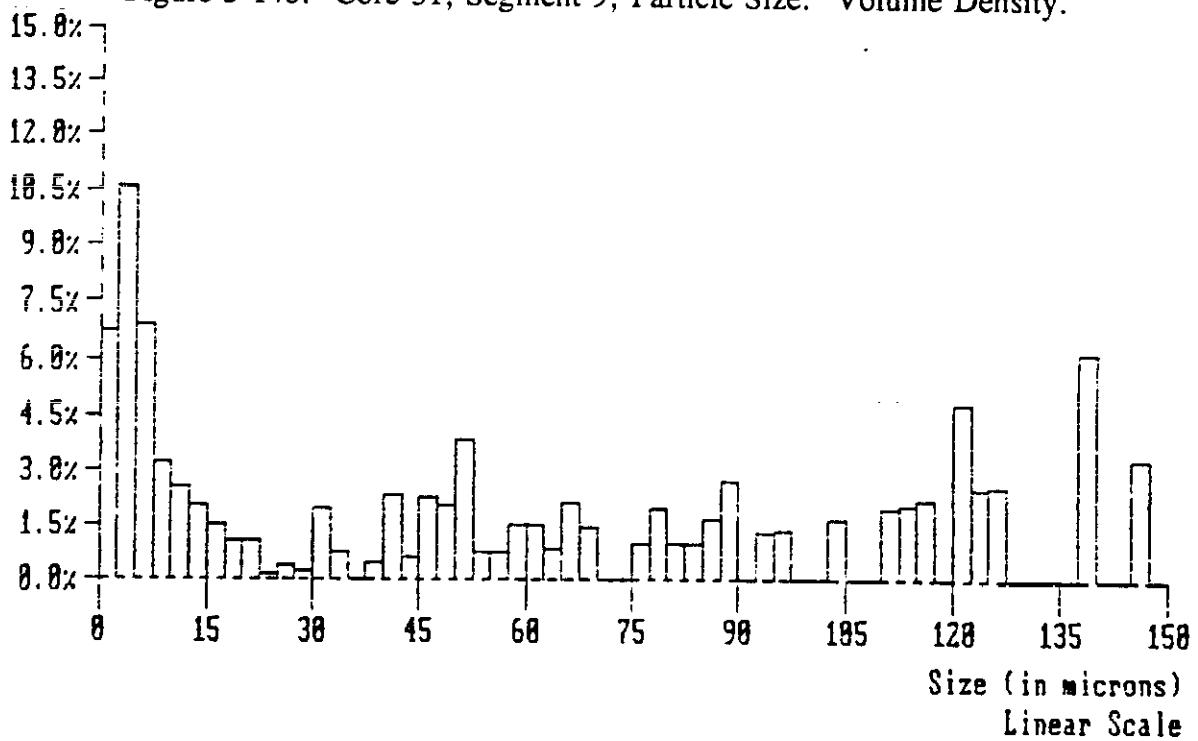


Table 5-15. Grab Sample Physical Properties Summary.

Property	Grab sample #1	Grab sample #2	Grab sample #3	Average value
Specific gravity	1.036	1.038	1.033	1.036
pH	11.57	11.59	11.78	11.65
Wt% H ₂ O	92.70	92.90	92.90	92.83
Wt% H ₂ O (TGA)	91.17	91.73	90.24	91.05
Settled solids (vol%)	< 1%	< 1%	< 1%	< 1%

The grab samples were clear yellow liquids with no particulate.

Table 5-16. Physical Properties Summary.

Property		Sample	
		Core 31, segment 2	Core 31, segment 8
Settled solids (vol%)	As-Received	100%	100%
Wt% Solids		22.4	29.3
Wt% Undissolved solids		19.0	25.4
Density (g/mL)		1.19	1.28
Centrifuged samples			
Vol%	1 hour at 1,000 gravities	65.8	71.9
Wt%		67.3	75.9
Centrifuged supernate density (g/mL)		1.07	1.10
Centrifuged solid density (g/mL)		1.22	1.34

No settling was observed in the as-received segment samples over a period of three days, and there was no standing liquid obtained from the samples. Two dilutions of 1 to 1 and 3 to 1 water to sample ratios, respectively were prepared, and the volume-percent settled solids for each of the dilutions are plotted as a function of settling time.

The 1 to 1 dilution for segment 2 reaches a final volume percent settled solids of 85 to 87 percent. Settling is observed throughout the three-day period, but the majority of the settling is observed in the first 10 hours. The 3 to 1 dilution reaches a final volume-percent settled solids of approximately 52 percent. Again, settling is observed over three days, and the first 10 hours is when the majority of the solids settle. Qualitatively, the settling behavior for both sample dilutions is a steep, nearly linear relationship between the initial

fluidization of the material and the first 10 hours of settling. After that, the final 10 percent of the suspended solids take up almost the rest of the time settling in a long, gradual decline, before coming to equilibrium.

The 1 to 1 and 3 to 1 dilutions for segment 4 were compromised by drying the sample before its assay. However, some observations and contrasts with the other samples are appropriate. Settling is mostly completed after 3 to 4 hours, and is complete after 10 hours. This is in sharp contrast to the other samples for which there is a long, asymptotic-like settling behavior observed for a substantial portion of the suspended solids (10 to 15 percent), after the initial settling phase. This behavior is suggestive of a colloid or gel for segments 2 and 8. In contrast, segment 4 appears to be a collection of discrete particles with no interaction between them.

The 1 to 1 dilution for segment 8 reaches a final volume-percent settled solids of about 80 percent (see Figure 5-17a). Settling is observed throughout the three-day period, but the majority of the settling is observed in the first 10 hours. The 3 to 1 dilution reaches a final volume-percent settled solids of approximately 40 percent. Again, settling is observed over three days and the first 10 hours is when the majority of the solids settle. Qualitatively, the settling behavior for both sample dilutions is a shallow, nearly linear relationship between the initial fluidization of the material and the first 10 hours of settling. The slope of this line is much more gradual than that of segment 2 for the corresponding dilutions. After the first 10 hours, the final 15 percent of the suspended solids take up almost the rest of the time settling in a long, gradual decline before coming to equilibrium. Table 5-17 summarizes the settling behavior for the samples investigated. Figures 5-15a through 5-17b illustrate the setting behavior over time.

Table 5-17. Settling Comparison for 1 to 1 and 3 to 1 dilutions for Core 31 Segments 2, 4, and 8.

Analyte	Segment 2		Segment 4		Segment 8	
Dilution: water to sample	1:1	3:1	1:1	3:1	1:1	3:1
Final volume % solids	87	52	22	22	80	40

5.4 ANALYTICAL RESULTS--ENERGETICS

TGA and DSC were performed on subsegment and core-composite material from tank 241-T-111. These two thermal analysis techniques are used to determine the thermal stability or reactivity of a material. In DSC analysis, heat flow over and above the usual heat capacity of the substance is measured while the substance is exposed to a linear increase in temperature, i.e., the change in temperature, divided by the time elapsed is constant ($dT/dt = \text{constant}$). While the substance is being heated, a cover gas (usually air or N_2) is passed over the waste material to remove any gases being released. The onset temperature

Figure 5-15a. Settling Rate Data for Tank 241-T-111 Core 31, Segment 2, 1 to 1 Dilution.

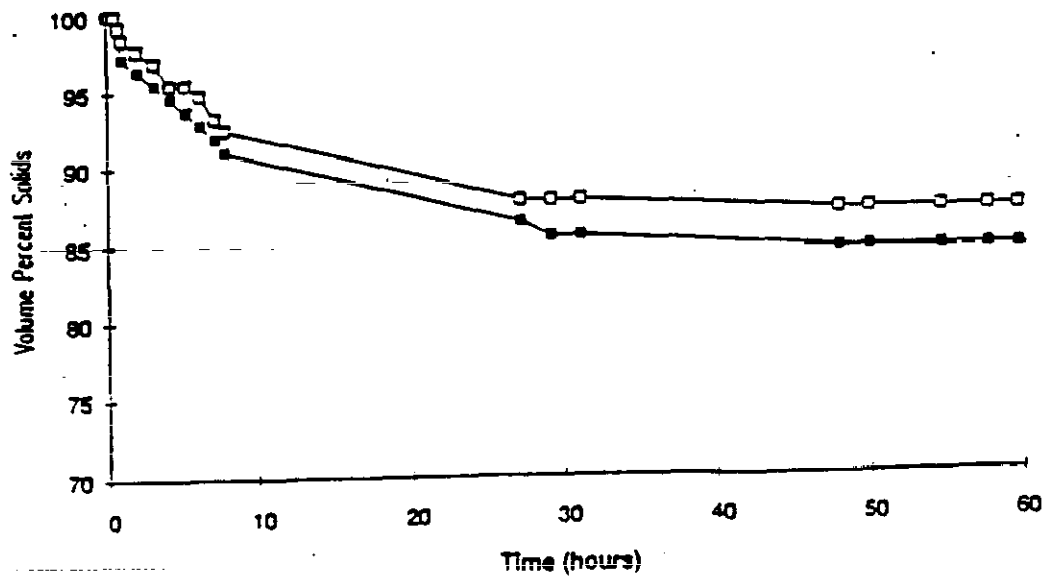


Figure 5-15b. Settling Rate Data for Tank 241-T-111 Core 31, Segment 2, 3 to 1 Dilution.

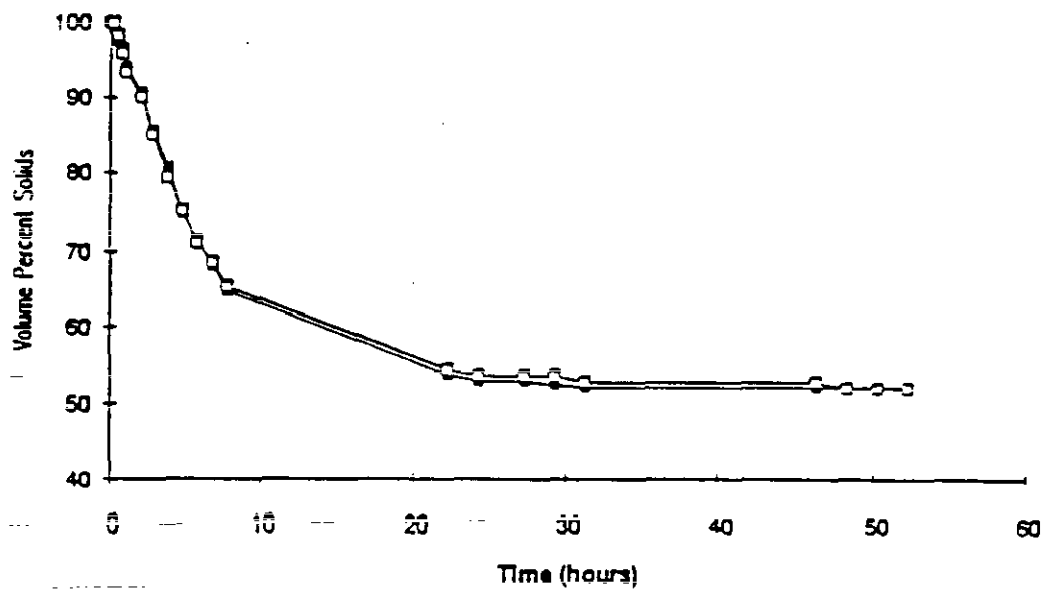


Figure 5-16a. Settling Rate Data for Tank 241-T-111 Core 31, Segment 4, 1 to 1 Dilution.

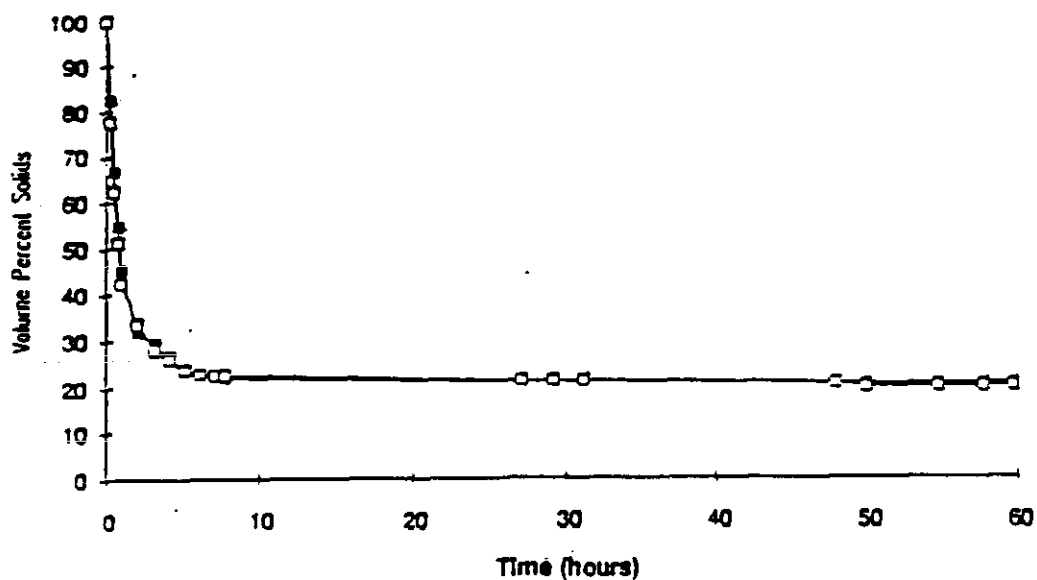


Figure 5-16b. Settling Rate Data for Tank 241-T-111 Core 31, Segment 4, 3 to 1 Dilution.

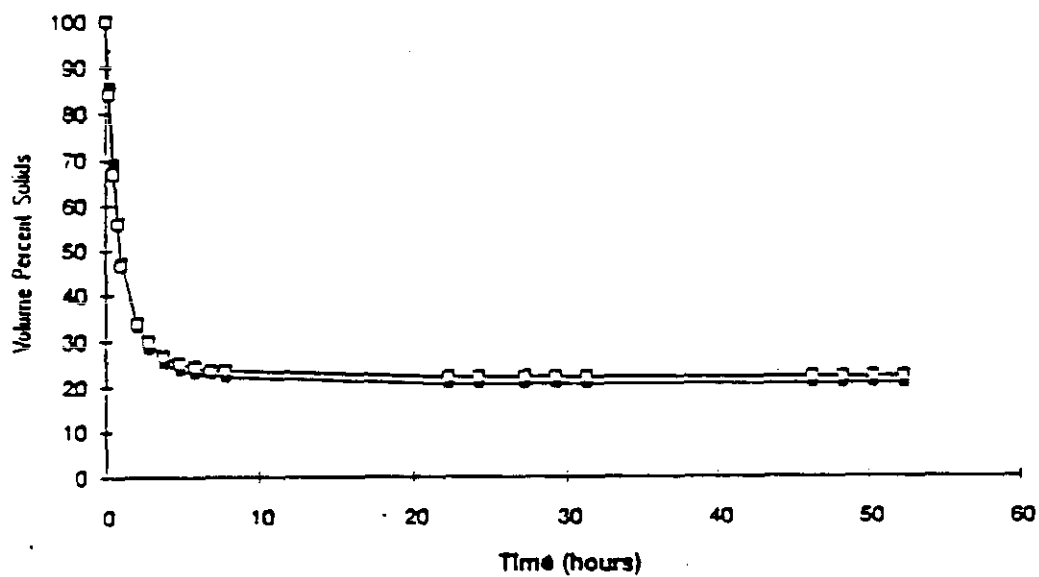


Figure 5-17a. Settling Rate Data for Tank 241-T-111 Core 31, Segment 8, 1 to 1 Dilution.

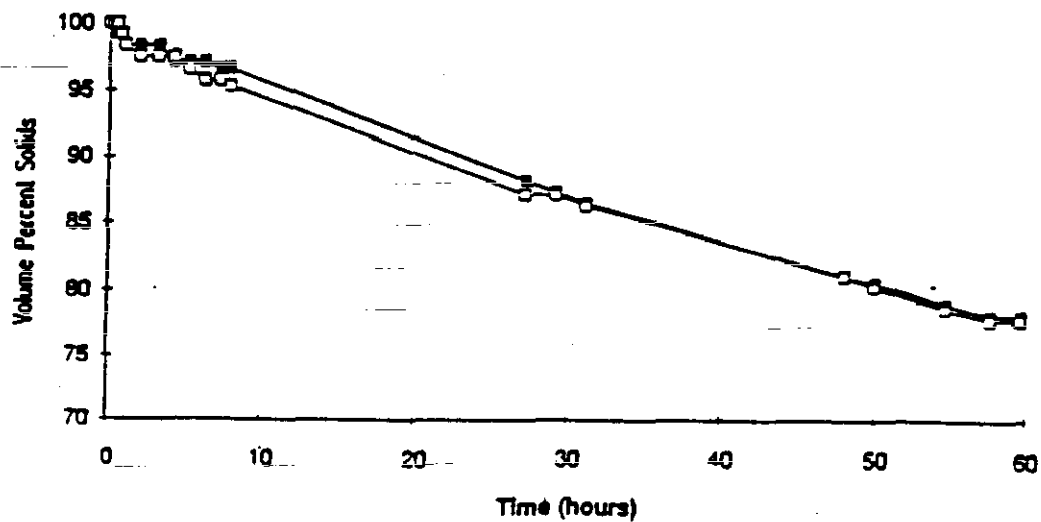
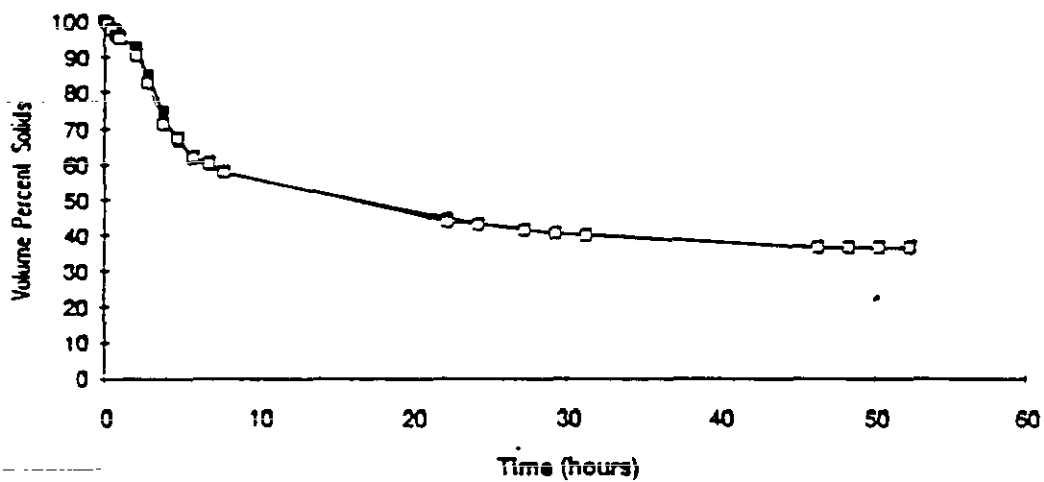


Figure 5-17b. Settling Rate Data for Tank 241-T-111 Core 31, Segment 8, 3 to 1 Dilution.



for an endothermic or exothermic event on a DSC is determined graphically. The endpoints of the event are determined and a line is drawn between them to establish a base. A line tangent to the initial side of the event is drawn until it intersects the base. From that point of intersection, a vertical line is constructed to the temperature scale at the bottom of the DSC curve. That temperature is the onset temperature of the event.

TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. The X-axis is representative of the running time of the analysis as well as the temperature increase of the sample during analysis. The Y-axis represents the weight percent of the sample and is effectively unitless. As with the DSC, a cover gas is passed over the sample during heating. Any decrease in the weight percent of the sample represents a loss of gaseous matter from the sample either through evaporation or a reaction that forms gas phase products.

DSC is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points, and solid-solid transition temperatures. TGA is used to measure thermal decomposition temperatures, water content, and reaction temperatures. The two methods often provide complementary information.

5.4.1 Remarks on the Interpretation of Differential Scanning Calorimetry/Thermogravimetric Analysis Data

Tables 5-18, 5-19, and 5-20 summarize the results of the thermal analyses performed. Although DSC assays were performed on the grab samples, the results were uniformly negative (i.e. no exotherms were observed). Where exotherms were observed, there are two significant features seen on the DSC and one on the TGA plots. The endothermic DSC event overlaps the area where the substantial majority of the sample mass is lost as recorded by the TGA, suggesting that this endotherm is a result of the evaporation of water from the sample. The values presented in the tables may not exactly match the values derived from the DSC and TGA plots, especially exotherm magnitudes. This is because interpreting these semi-quantitative analyses requires considerable experience and judgement on the part of the analyst, and differences in perception and judgement between readers can be expected in a semi-quantitative analysis. Appendix A contains copies of all of the DSC and TGA traces and results.

Additional physical properties work was performed to expand the understanding of the exothermic behavior of some of the segments (WHC 1994, Delegard 1994). Table 5-21 presents additional energetics results for core 33, segments 1 and 2. These samples were dried under a vacuum at 60 °C before analysis, using either air or nitrogen as a cover gas. Even after drying, the samples retained 10 to 12 weight percent water. Table 5-22 presents a brief summary of the analytical results for the average sample properties as-received after centrifuging samples from core 31, segments 3 and 7, and core 33, segments 1 and 7, at 500 gravities for 113 hours.

5.4.2 General Comments on the Differential Scanning Calorimetry/ Thermogravimetric Analysis Behavior of the Samples

The first transition in each sample is endothermic, begins at the lower temperature limit of the analysis (30 °C), and essentially is complete between 140 and 180 °C. The most likely phenomenon occurring in this region is the release of the bulk and interstitial water in the core sample material. The endotherms exhibited in this region are substantial (typically in excess of 1,000 J/g). These values are per gram of wet sample. If divided by the mass fraction lost during analysis, they range from 1,600 to 1,900 J/g (dry) and correspond roughly with the heat of vaporization of water (2,260 J/g). The TGA water content corresponds reasonably well with the water loss observed in a gravimetric weight percent solids determination; however, the gravimetric weight percent water assay is consistently lower.

Table 5-18. Percent Water Analyses Results
from Tank 241-T-111.

Sample I.D.	Core 31 average gravimetric Wt% loss	Core 31 average TGA transition 1 Wt% loss	Core 33 average gravimetric Wt% loss	Core 33 average TGA transition 1 Wt% loss
Segment 1	80.3	87.0	80.4	NM
Segment 2	82.4	87.0	85.7	80.6
Segment 3	86.0	85.0	81.8	88.5
Segment 4	77.3	82.8	79.9	89.5
Segment 5	80.9	88.0	78.2	88.8
Segment 6	No sample	No sample	75.8	84.4
Segment 7	76.8	84.8	71.7	85.8
Segment 8	76.6	85.6	75.4	84.8
Segment 9	75.8	71.0	76.0	85.2
Segment 9B	70.4	72.1	NA	NA
Composite 1	74.6	73.3	76.5	81.6
Composite 2	75.9	70.2	77.1	80.8

Table 5-19. Differential Scanning Calorimetry Energetics Results from Tank 241-T-111, Core 31 (wet basis).

Core sample 31	Transition 1			Transition 2		
	Range (°C)	Avg. onset (°C)	ΔH range (J/g)	Range (°C)	Avg. onset (°C)	ΔH range (J/g)
Segment 1	43 - 141	43	1,088 to 1,406	200 - 387	200	-259 to -273
Segment 2	46 - 149	46	1,108 to 1,643	200 - 398	225	-256 to -264
Segment 3	45 - 160	50	1,210 to 1,233	195 - 405	198	-263 to -448
Segment 4	44 - 165	44	1,235	200 - 390	200	-55.7
Segment 5	NR	--	Endotherm NR	NA	NA	No Exotherm
Segment 6	NA	--	---	---	--	NA
Segment 7	50 - 164	50	1,488	165 - 400	NA	No Exotherm
Segment 8	50 - 153	50	1,534	154 - 400	NA	No Exotherm
Segment 9	61 - 158	61	1,437	159 - 400	NA	No Exotherm
Composite 1	NR	--	Endotherm NR	256 - 339	257	-23.6 to -37.0
Composite 2	NR	--	Endotherm NR	260 - 334	262	-18.5 to -22.9

NOTE: To convert from J to cal, divide by 4.18.

NOTE: Negative ΔH indicates an exotherm.

NA = Not applicable.

NR = Not resolved.

Table 5-20. Differential Scanning Calorimetry Energetics Results from Tank 241-T-111, Core 33 (wet basis).

Core Sample	Transition 1			Transition 2		
	Range (°C)	Avg. onset (°C)	ΔH range (J/g)	Range (°C)	Avg. onset (°C)	ΔH range (J/g)
Segment 1	49 - 168	49	958 to 1,604	168 - 374	184	-218 to -293
Segment 2	45 - 179	50	1,346 to 1,496	168 - 438	174	-454 to -645
Segment 3	NA	--	Endotherm NR	237 - 400	237	-49.3
Segment 4	NA	--	Endotherm NR	NA	--	No Exotherm
Segment 5	NA	--	Endotherm NR	NA	--	No Exotherm
Segment 6	NA	--	Endotherm NR	NA	--	No Exotherm
Segment 7	NA	--	Endotherm NR	NA	--	No Exotherm
Segment 8	NA	--	Endotherm NR	NA	--	No Exotherm
Segment 9	NA	--	Endotherm NR	NA	--	No Exotherm
Composite 1	NA	--	Endotherm NR	NA	--	No Exotherm
Composite 2	NA	--	Endotherm NR	NA	--	No Exotherm

NOTE: To convert from J to cal, divide by 4.18.

NOTE: Negative ΔH indicates an exotherm.

NA = Not applicable.

NR = Not resolved.

Table 5-21. Differential Scanning Calorimetry Energetics Results
from Tank 241-T-111, Core 33 (dry basis).

Laboratory-core sample-air/N ₂	Transition 1			Transition 2		
	Range (°C)	Avg. onset (°C)	ΔH range (J/g)	Range (°C)	Avg. onset (°C)	ΔH range (J/g)
222-S core 33, seg. 1, Air	---	---	NR--Dried	158 - 405	NR	-1,857 to -1,882
222-S core 33, seg. 2, Air	---	---	NR--Dried	130 - 425	NR	-251 to -269
222-S core, 33 seg. 2, N ₂	---	---	NR--Dried	130 - 430	NR	-288 to -309
222-S core 33, seg. 2, N ₂	---	---	NR--Dried	128 - 418	NR	-180 to -187
222-S core 33, seg. 2, N ₂	---	---	NR--Dried	123 - 421	NR	-163 to -175
222-S core 33, seg. 2, N ₂	---	---	NR--Dried	121 - 438	NR	-336
325 core 33, seg. 2, N ₂	---	---	NR--Dried	107 - 394	199	-836 to -898

NOTE: To convert from J to cal, divide by 4.18.

NOTE: Negative ΔH indicates an exotherm.

NA = Not applicable.

NR = Not resolved.

Table 5-22. Additional Segment-level Physical Properties Measurements (1994).

Analyte	Core 31, segment 3		Core 31, segment 7		Core 33, segment 1		Core 33, segment 7	
	As- received	Centrifuged	As- received	Centrifuged	As- received	Centrifuged	As- received	Centrifuged
Gravimetric water (%)	79.53	64.96	74.72	62.06	79.56	65.49	74.07	59.95
TGA (%)	76.72	55.36	74.06	55.83	78.08	51.37	78.1	45.15
Density (g/mL)	1.24	1.09	1.19	1.20	1.16	1.19	1.20	1.29
ΔH exotherm range (J/g)	-112 to -191	-465.3 to -546.9	-10.2 to -33.1	0	-249 to -254	-822.4 to -838.1	-37.5 to -41.4	0

When there is a second transition it is usually substantial and the energetic behavior is readily quantifiable in all of the samples analyzed where exotherms are observed. Any weight loss in the second transition region (generally temperatures above 200° C) was not readily quantifiable, whether exotherms were observed or not. The results for the samples from segments 1, 2, and 3, which are from the upper portion of the tank, indicate significant differences in thermal behavior compared to other samples from deeper in the tank, further suggesting a difference in waste type. In addition, because of the observed exothermic behavior for the top 3 segments from cores 31 and 33 and the results from Baldwin (1994), the present TOC assay is not considered capable of measuring the TOC in the waste. Resolution of the actual magnitude of the exotherm, its reaction mechanism, and speciation of the fuel is still continuing.

5.5 OVERALL ANALYTICAL DATA SUMMARY

Several characterization and safety issues are defined by certain bulk amounts or weight percent of a given analyte. Table 5-23 presents the nominal concentration and calculated bulk amounts of the analytes in the waste matrix. The gross waste inventory in the tank is estimated to be 2,171,000 kg wet solid. It is assumed that by the publication of this report, no drainable liquid will remain in the tank. Appendix C presents the data, assumptions, and calculations used to determine the following values.

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

GROUP	Process history ¹ (Agnew 1994)	TRAC (Jungfleisch 1984)	Analytical data range (most quantitative assay)	1992 cores 31 and 33 average	Tank inventory based on 1992 core data
CATIONS	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	kgs
Be.f	---	---	< DL	< DL	< 0.22
B.a	---	---	23.4 - 32.2	28	60.8
Na.f	74,200	0	33,900 - 39,800	37,000	80,300
Mg.a	---	---	290 - 479	377	820
Al.f	0	0	459 - 693	570	1,240
Si.f	2,800	0	5,410 - 5,960	5,670	12,300
P.f	(as PO_4^{3-})	(as PO_4^{3-})	9,070 - 11,600	10,400	22,600
S.f	(as SO_4^{2-})	(as SO_4^{2-})	1,080 - 1,350	1,230	2,660
K.a	3,700	0	1,020 - 1,210	1,140	2,460
Ca.f	0	0	2,050 - 2,760	2,420	5,260
Ti.f	---	---	22.3 - 72.9	48	104
V.f	---	---	12.1 - 16.5	14.5	31.5
Cr.a	33	719	1,840 - 2,140	1,980	4,290
Mn.a	90	507	6,140 - 6,710	6,330	13,700
Fe.f	9,400	10,300	15,900 - 20,500	18,500	40,200
Ni.a	0	0	108 - 151	132	285
Co.f	---	---	10.1 - 13.3	4.5	9
Cu.f	---	---	22.1 - 36.3	29.3	63
Zn.f	---	---	104 - 110	106	231
As.a	---	---	< DL	< 3.3	< 7.2
Se.a	---	---	< DL	< 1.5	< 3.3
Sr.f	0	---	280 - 334	300	651
Zr.f	0	0	4	NR	---
Ag.f	---	0	37.1 - 221	130	278
Cd.f	---	---	6.42 - 10.7	5.8	12.6
Sn.a	---	---	1.61 - 4.21	2.5	5.4
Sb.a	---	---	22.6 - 36.5	31.4	70
Ba.a	---	0	57.0 - 87.3	69	150
La.a	12,700	512	3,620 - 4,890	4,220	9,160

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

GROUP	Process history ¹ (Agnew 1994)	TRAC (Jungfleisch 1984)	Analytical data range (most quantitative assay)	1992 cores 31 and 33 average	Tank inventory based on 1992 core data
CATIONS	μg/g	μg/g	μg/g	μg/g	kgs
Ce.a	0	0	28.6 - 37.8	33.7	73.2
Hg.CVAA	—	—	1.08 - 1.83	1.43	3.1
Pb.f	0	0	267 - 484	365	792
Bi.a	23,200	963,000	23,300 - 28,500	26,000	56,300
U.LF	140	—	1,950 - 5,200	3,550	7,700
RADIONUCLIDES					
Analyte	μCi/g	μCi/g	μCi/g	μCi/g	Ci
Total Alpha	—	—	0.166 - 0.649	0.368	NA
Total Beta	—	—	8.83 - 21.5	15.1	NA
²⁴¹ Am	—	0.0092	0.0382 - 0.0478	0.0425	92.4
^{239/240} Pu	0.009	0.055	0.134 - 0.628	0.304	660
¹³⁷ Cs	0.086	0	0.103 - 0.237	0.166	360
⁹⁹ Tc	—	0	0.00473 - 0.0114	0.0079	17.2
⁵⁹ Ni	—	—	3.35E-05 - 9.4E-05	5.04E-05	0.11
⁶³ Ni	—	—	0.0036 - 0.011	0.0057	12.4
⁹⁰ Sr	0.176	1.84	3.43 - 7.43	5.41	11,800
¹⁴ C	—	0	< DL	< DL	—
³ H	—	—	< DL	< DL	—
ANIONS					
Analyte	μg/g	μg/g	μg/g	μg/g	kgs
OH ⁻	8,900	15,700	3,300 - 6,000 ²	—	—
NH ₃	—	—	< DL	< DL	—
F ⁻	24,600	0	1,370 - 3,130	2,300	4,990
Cl ⁻	0	0	400 - 500	450	977
NO ₂ ⁻	0	0	525 - 952	793	1,720
NO ₃ ⁻	51,600	0	36,900 - 44,300	41,300	89,660
CO ₃ ⁻	0	0	as TIC	812	1,760
Total PO ₄ ⁻³	35,400	438,000	27,800 - 35,500	31,900	70,100

Table 5-23. Overall Data Summary and Inventory Estimates. (3 pages)

GROUP	Process history ¹ (Agnew 1994)	TRAC (Jungfleisch 1984)	Analytical data range (most quantitative assay)	1992 cores 31 and 33 average	Tank inventory based on 1992 core data
CATIONS	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	kgs
ANIONS					
SO_4^{2-}	1,450	0	3,290 - 3,690	3,680	7,990
CARBON-BEARING SPECIES					
Analyte	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
TOC	2,000	---	2,000 - 3,990	3,120	6,770
TIC	---	0	650 - 950	812	1,760
$\text{C}_2\text{H}_3\text{O}_2^-$	0	0	NM	---	---
$\text{C}_2\text{O}_4^{2-}$	7,200	---	NM	---	---
$\text{C}_6\text{H}_5\text{O}_7^-$	0	0	NM	---	---
$\text{Fe}(\text{CN})_6^{4-}$	0	0	NM	---	---
EDTA	0	0	NM	---	---
HEDTA	0	0	NM	---	---
PHYSICAL PROPERTIES					
Wt% Water (Grav.)	72.5%	---	74.6 - 77.1	76.0%	1.65E+06 kg
Wt% Water (TGA)	72.5%	---	70.2 - 81.6	76.5%	1.66E+06 kg
Bulk Density (g/cm^3)	1.21	1.8	1.19 - 1.28	1.24	---
Supernatant Density (g/cm^3)	1.058	---	1.033 - 1.038	1.036	---

¹Process history estimates are determined using a simple linear combination based on the proportions of 2C and 224 waste contributed.

²OH⁻ is estimated from mass and charge imbalance.

6.0 INTERPRETATION OF ANALYTICAL RESULTS

Tank 241-T-111 had a relatively straightforward process history as documented in the transfer records. It received very few major types of waste that were likely to deposit solids during its operating history. The waste types, in chronological order, were as follows:

- 2C waste
- 224 Waste
- Decontamination streams from T Plant (221-T).

The purpose of this section is to attempt to identify and reconcile the location of the tank waste solids, thereby estimate the tank inventory for various analytes of importance.

The waste profile was identified by examining the available segment level assays for analytes or characteristics distinct to the waste types that were disposed in the tank, and then combining that information with what is known regarding the tank's process history. The first waste placed in the tank through the cascade inlet from tank 241-T-110 was 2C waste. Study of the process stream compositions indicates that this waste would be comparatively high in bismuth and phosphate in addition to the ubiquitous sodium, nitrate, and iron found in nearly all waste types. Elevated levels of fluoride, chromium, and sulfate are also expected. Qualitatively, the waste has been observed to be a gelatinous material. Anecdotal reports during this time also indicate that the cascade lines may have clogged in tank 241-T-111 and in other tank cascades receiving this type of waste. The 2C solids volume was measured to be between 723,000 L (191,000 gal) and 931,000 (246,000 gal) in 1953 (Anderson 1990).

The tank then received 224 waste. The solids from this waste are high in manganese, lanthanum, and fluoride, however, the 224 waste may have been combined with the 2C waste before being discharged to the tank. The estimated solids volume contribution for this waste type in tank 241-T-111 at the end of bismuth phosphate production in T Plant was between 594,000 and 802,000 L (157,000 and 212,000 gal).

The last major waste type disposed in the tank was T-Plant decontamination waste. Much of the physical and chemical composition of this waste is unknown because it was a catch-all, consisting of various process residues, unused stock solutions, and aqueous decontamination solutions that contained surfactants (such as Turco[®]). Few predictions can be made from

[®]Turco is a registered trademark of Turco Products, Inc.

studying the historical process flowsheets in this case. Lack of analytical data and/or transfer records with regard to T-Plant effluents later in tank 241-T-111's service life are a great source of uncertainty regarding the waste near the surface. One observation is that slightly higher TOC values may be anticipated near the surface because of the use of detergents in the decontamination waste stream and its chronological discharge sequence with respect to the other wastes. This waste stream is estimated to occupy the top segment of waste, or 198,000 L (53,000 gal). The sum of the wastes would range between 1.52 million L and 1.93 million L (401,000 gal and 511,000 gallons), well within the reported range described in Anderson (1990). This waste volume would measure between 389.4 to 491.0 cm (153.3 to 193.3 in), measured from the centerline and distributed evenly across the tank. The present surveillance level status is almost exactly in the middle of this range 1.73 million L (456,000 gal) and $440.2 \text{ cm} \pm 1.3 \text{ cm}$ ($173.3 \pm 0.5 \text{ in}$) (Rios 1994).

Two common characteristics of all these waste types are high water content and relatively low activity. None of the waste streams disposed in this tank were concentrated through the evaporator; therefore, the waste tank would not have any salt cake. The uranium, plutonium, and fission product content of these wastes are uniformly low.

6.1 Review of the Analyte Profiles

The following conclusions are drawn from review of the available composite and segment analyses presented in Section 5, and the historical information presented in Section 2.

Core 31

The chemical analyses of core 31 indicate there are at least two primary types of material in distinct layers in the tank. The DSC traces for segments 1, 2, and 3 show exotherms far out of proportion to the measured organic content in the wastes. The temperature range where the wastes begin to show reactions are from 170 to 400° C, but do not appear to be self-sustaining. Instead, the overall energy profile is highly endothermic, probably caused by the large amount of bound water (70 to 80 weight percent) that is evaporated from the sample before a reaction is initiated. The overall physical and chemical properties of the waste in the tank roughly correspond to the expected behavior and composition of 2C waste, with large quantities of bismuth, iron, and phosphorous, and no exothermic behavior. But, there is a substantial contribution of manganese and lanthanum in the composites. However, these analytes can be found in 224 waste, and historical data indicates that 224 waste was added to tank 241-T-111 later in its service life. Therefore, the analytical and historical data correspond reasonably well, except for the anomalous energetic results. At this time, there is no adequate explanation for the observed exotherms in the upper segments of tank 241-T-111 waste.

Core 32

Every segment of core 32 was compromised during the sampling process in some fashion. When the sampler operated properly, liquids were the only material recovered from this core. However, the sampler valve failed repeatedly. Photographs taken before and after the sampling event reveal that there was a plastic bag in the vicinity of the sampling area before coring operations. After core sampling, the bag can not be seen. It is surmised that the sampling drill string was obstructed by the bag during core sampling operations, causing the corruption of the samples. All samples from core 32 were rendered unusable or categorized as non-representative. Therefore, no assays were performed and no analytical results are reported.

Core 33

The chemical analyses of core 33 indicate there probably are three primary types of material in distinct layers in the tank. The extremely high manganese values in the top first segment of the tank are attributed to a combination of T-Plant decontamination waste (i.e. the result of a final process flush during the decontamination of T Plant) and 224-waste solids deposited late in the tank's service life. Proceeding deeper into the tank, beneath the first segment, the distribution of bismuth, manganese, lanthanum, and chromium in the composites and the analyte profiles from the homogenization results through segment 3 support the conclusion that this material is still 224 waste. Segment 3 itself is suspected to be a transition layer, containing the boundary between the 224 waste and the 2C waste, based on the exothermic behavior of the waste as a function of depth. The TOC analysis indicates moderate amounts of residual organics in the waste.

Cesium - 137 concentrations between the core composites vary within a factor of two, and the variation in the ^{241}Am is less than 13 percent. But the change in concentration as a function of depth for each of these analytes is much more significant. Both analyte profiles show a decreasing trend as a function of depth in core 33. The ^{137}Cs concentration decreases by more than a factor of 30 over the depth of the tank, and ^{241}Am decreases by a factor of 10 through segment 7 before rebounding somewhat in segment 9. The ^{137}Cs and ^{241}Am concentrations as a function of depth in core 33 show profiles consistent with the wastes believed to be associated with the segments: low overall ^{137}Cs and ^{241}Am values. But the relative radionuclide concentrations for the suspected 224 and T-Plant decontamination wastes are higher than the 2C wastes. The ^{90}Sr concentration is also low, and ^{90}Sr is similar to ^{137}Cs in the magnitude of the change in concentration and as a function of location for both cores. However, there are no high radionuclide values anywhere in the tank, and the tank temperature further confirms the tank's low radionuclide content.

Lanthanum and phosphate/phosphorus demonstrate an increasing concentration profile as a function of depth. Between segments 1 and 3 and 3 and 5 there is an abrupt change in the concentrations of calcium, chromium, manganese, bismuth, lanthanum, and phosphorous, although after segment 5, the analyte concentrations tend to plateau, mirroring the behavior

of the radionuclides. In addition, the DSC traces for segments 1, 2, and 3 show exotherms far out of proportion to the measured organic content in the waste. The temperatures where the wastes begin to show reactions range from 170 to 400 °C, but these reactions do not appear to be self-sustaining. Instead, the overall energy release is highly endothermic, probably from the large amount of bound water that is evaporated from the sample before a reaction is initiated.

In conclusion, the physical and chemical properties of the waste in the bottom half of the tank correspond to the expected behavior and composition of 2C waste, and the upper half of the tank is suspected to be 224 waste with a high manganese layer located in segment 1. Therefore, the analytical and historical data correspond well with the historical fill pattern, except for the anomalous energetic results. Presently there is no adequate explanation for the observed exotherms in the upper segments of tank 241-T-111 waste.

6.1.1 Entrance, Exit, and Mixing Effects on Analyte Distribution

Figure 5-1 shows an elevation and plan view of where the core samples were taken. Important items to note are the arrangement and location of the risers and cascade lines (inlet and outlet). Their configuration can have a substantial impact on the distribution of waste in the tank, and observations with regard to sampling. However, the waste entrance and exit points for the tank over its service life are not well documented, therefore the spatial relationship and proximity to the sample risers is not known. The decant "float and flex" pump contained a 6.1-m (20-ft) section of flexible hose that could traverse a relatively wide area under and around the pumpout riser. The cascade fill line where 2C waste entered the tank is closer to the core 33 sample point than to the core 31 sample point. There was very limited discharge/transfer traffic from the other separations plants within T farm or with the tank farms as a whole, so no highly enriched layer of radioactive material is expected to lie on top of the waste as has been observed in other tanks sampled. However, the relative concentration of radionuclides is observed to be higher in the upper portions of the tank.

As new wastes entered the tank and were distributed across it, the material under and around the tank pumpout could have been disturbed, and occasionally solids could have been transferred, in behavior similar to the last in, first out principle. It is believed that the material beneath the waste inlets, the cascades and perhaps a riser, would have been disturbed initially, but over time, large stratified layers resistant to mixing eventually would have built up. No deliberate mixing of the wastes was performed; therefore, where segment-level data is available, distinctions between waste types can be made. Some of the larger particulate materials discharged to the tank initially may have settled out near the inlet because they were not as flocculent or as easily suspended as some of the other solids. This settling behavior may have provided a slight degree of separation. Therefore, the influence of the waste inlet and outlet locations can provide insight to the analyte distribution and waste profiles between cores 31 and 33. These factors may account for the nominally observed lateral heterogeneity between cores 31 and 33; however, it must be noted that this lateral

heterogeneity is very slight and there may be several other factors contributing to this observation.

6.1.2 Waste Profile

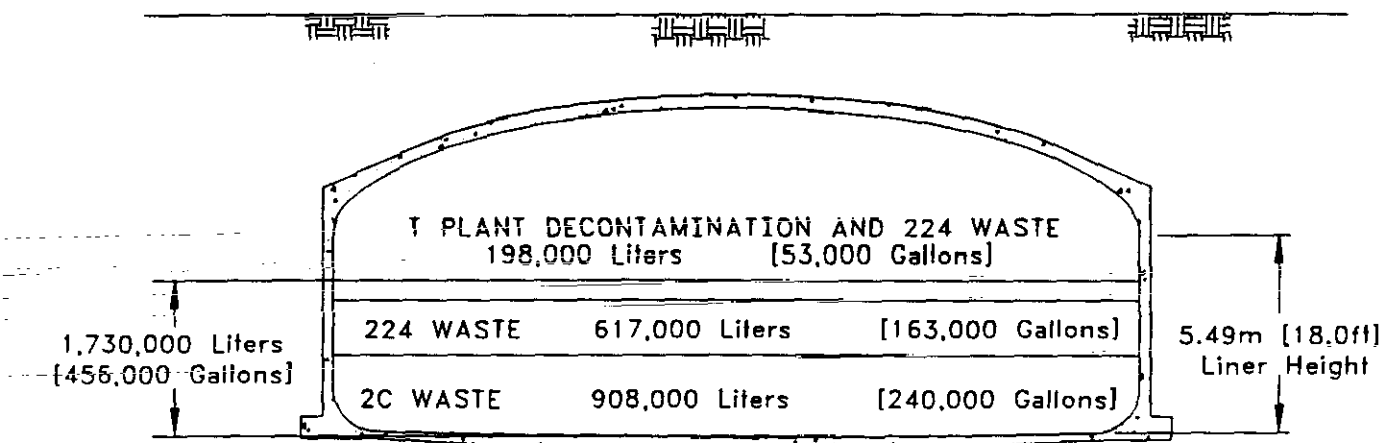
Given the historical and analytical data, it is possible to develop a rough representation of the wastes as they presently are configured in tank 241-T-111. Figure 6-1 shows a representation of the overall waste profile of tank 241-T-111 and the assumed volumes, boundaries, and positions of the various individual layers as they are believed to exist.

6.2 WASTE SUMMARY AND CONDITIONS

Historically, 241-T-111 was a non-watch list single-shell tank with no previous indication of a potential safety issues. All waste receipts were 2C waste, 224 waste, or residues from the T-Plant cleanout. Flowsheet records indicate that small amounts of oxalate are present in 224 waste (at 0.028 M) (Schneider 1951). As a result of DSC measurements, tank 241-T-111 was added to the Organic Watch List on March 4, 1994 and further concerns have been raised about the tank's integrity.

When tank 241-T-111 was sampled, normal paraffin hydrocarbon was used as a hydrostatic fluid, presenting a potential contamination source and bias for the DSC. However, when taking the first segment, no normal paraffin hydrocarbon was used, and sample recovery for the other segments in the cores was excellent. No voids or separable liquid layer were observed on extrusion of the samples, and results from the gas chromatograph/mass spectrometer for normal paraffin hydrocarbons components showed only trace amounts, precluding significant sample contamination from the hydrostatic head fluid. Furthermore, experiments have demonstrated that normal paraffin hydrocarbons steam distill away before reaching the reaction temperatures observed in DSC assays of the samples. In the deeper segments and in the composites, core 31 demonstrates some activity, but not to the extent shown in the upper segments. In core 33, no significant exotherms are evident in the composite. The photographs of the extruded waste material demonstrated that all of the core samples were viscous, gel-like materials with very little free liquid, and that they held their shape relatively well. During the physical testing of the core 31, segment 2 solid sample, when the sample was centrifuged, there was some separation of free water from the gel. Later physical testing did show some separation of liquid from the waste matrix for segment 7, but again, no separable phase was observed in the liquid (Delegard 1994). In each case, the liquid did not appear to have any distinct layers, therefore no liquid organics are considered to contribute to this waste matrix. In addition, although historically there have been problems with both volatile and semi-volatile organics analysis methods and

Figure 6-1. Waste Profile of Tank 241-T-111.



- Dished bottom and tank layer 1: 2C, 908,000 L (240,000 gal)
- Tank layer 2: 224 waste, 617,000 L (163,000 gal)
- Tank layer 3: T-Plant decontamination and 224 wastes, 198,000 L (53,000 gal)

holding times on these samples, all of the results from these assays show levels of these compounds below EPA contract laboratory procedure quantitation limits.

This information, coupled with data from the organic vapor monitor taken during the recent liquid sample effort (Appendix C), supports the contention that there are no substantial liquid organics in the tank. The organic vapor monitor results from the vapor space of tank 241-T-111 show a reading of 9.2 parts per million over a three minute monitoring interval: a detectable concentration, but far below the established safety criterion of 20 percent of the lower flammability limit. This result from the organic vapor monitor is not unexpected because small quantities of ammonia were observed in the grab samples and historically ammonia was used as a process chemical in the tank wastes. Over time, the ammonia is believed to have dissipated slowly to the levels observed today. It is also believed that the vapor space of this tank is relatively homogenous, and therefore, large concentration gradients for organics and other materials in the vapor space are not plausible. Differences of 2 to 10 times are within the realm of possibility (10 times being an extreme). Factors higher than that are not considered credible. For comparison, the vapor space of tank 241-C-103, which has a known organic liquid layer, was found to be 200 ppm. Furthermore, a simplified modeling scenario of tank 241-T-111 shows that if normal-paraffin-hydrocarbon-type liquid organics were present, they would be in much greater quantities than detected in the organic vapor monitor (see Appendix C).

To conclude, the waste in tank 241-T-111 is a complex material, primarily made up of water and organic and inorganic salts in a gel-like matrix. The insoluble solids are a mixture of phosphates, silicates, hydrated oxides, and hydroxides in combination with calcium, chromium, lanthanum, iron, bismuth, manganese, and uranium. The soluble analytes are primarily sodium, nitrate, sulfate, and fluoride. Phosphorous is nearly evenly divided between its soluble and insoluble forms. Substantial exotherms were detected in segments 1, 2, and 3, with the reactivity tailing off in segment 4 of core 31 and in segments 1, and 2, with the reactivity tailing off in segment 3 of core 33. The exotherms were reported to be similar in size and temperature range. It is important to note that no exotherms are observed until the sample has been dried and heated to approximately 180 °C. The exotherms themselves do not appear to be kinetically fast, thus the reaction may not be able to sustain itself without being thermally driven, as they are in the DSC apparatus. The organics present appear to be in the form of slightly soluble salts, contributing to a gel or sol-like structure. This behavior would be consistent with the very high moisture content observed in these tanks and with the types of organics historically indicated to be in the waste: aqueous decontamination solutions using surfactants, not normal paraffin hydrocarbons or TBP-type organics. This would help explain their low volatility and reactivity, their presence in the solid phase, and their relative absence in the liquids. These materials are carbon-bearing and will, if given enough impetus in the form of a thermal driver, react; however, the reaction does not appear rapid or self-sustaining, and will not occur without first removing the water from the gel. No other safety issue was found after critically reviewing the analytical, historical, and surveillance data.

Hypotheses Regarding Recent Behavior of 241-T-111

Recently, surveillance data indicated that there had been a relatively abrupt level change in tank 241-T-111 over the past 16 to 18 months. Approximately 2.5 cm (1 in) of gradual sludge growth was observed over the last eight-year period, followed by an approximately 4.1-cm (1.6-in) decrease in surface level over 16 months. This situation is cause for concern. Tank 241-T-111 is an assumed leaker, therefore there is the possibility that the tank has leaked again and waste is being lost to the environment. This is not an acceptable condition. *Tank Farm Operating Procedures* (Boyles 1992) specify that when a level drop in a tank is observed, the observation must be accompanied by certain actions, and that a leaking tank must be pumped to eliminate any remaining drainable liquid to prevent further environmental contamination. However, the observed level decrease was not large enough to trigger action past investigation under present guidelines (Boyles 1992), and it was not resolved that a leak was the only explanation for the observed behavior. In fact, there were observers who believed there was another safety issue requiring consideration involved. The final outcome of the review, however, was the decision to pump the liquid in the saltwell of tank 241-T-111 to tank 241-SY-102 (Jenkins and Engelman 1994). This section recaps some of the alternative explanations presented for the surveillance data and provides some of the strongest and weakest points of the arguments put forth.

- Tank is a "Re-leaker"

Evidence supporting this point of view is strong and it is one of the most favored explanations for the observed level drop. Tank integrity was questionable for a long period of time, and the tank was declared a leaker in 1984. Corrosion of tank liner is also evident from in-tank photographs. However, the change in surface level could be a localized phenomenon and in-tank photographs and observations of the extruded core material indicate that the waste is viscous and cohesive. Substantial damage to the tank liner and shell would be necessary for the evacuation of over 11,000 L (3,000 gal) of material and this condition is not indicated.

- Long Cycle Gas-Release Tank

This proposition is more speculative, but warrants consideration. Cyclic, gas generating behavior previously has been observed in Hanford-Site waste tanks. Specifically, tank 241-T-110, the cascade source for tank 241-T-111, is on the Gas-Generation Watch List. Viscous waste material, such as that observed in tank 241-T-111, also appears to be able to retain generated gases. However, overall tank information, specifically the analytical chemistry results, indicate tank conditions necessary for gas generation as currently understood (i.e. high radionuclide and complexant levels), do not exist.

- Intrinsic Waste Matrix Changes

This explanation is even more tenuous than the previous one, but again, the present understanding of the high-level waste tank matrices is very limited, so it is within the realm of possibility. Composition and structure of the waste (small particles, high water content, high ionic strength solution, and possible organic surfactants), may be similar to an emulsion. Over time, the low-level radiolytic action and thermal cycling of the waste may break down the colloidal characteristics of the matrix. The breakdown may be uneven, and there may even be a slurry-growth phase before the separation of the emulsion and the loss of free water through pinhole corrosion. However, that same degree of uncertainty regarding the content and structure of the waste matrix makes this hypothesis highly speculative without much more characterization information than currently exists.

- Structural Subsidence of the Waste

This particular condition may be a contributing factor to the observed behavior, since it is acknowledged that the overall configuration of the waste generally is not very well known (and that the surfaces of the waste beds can be highly irregular), however it is not considered the main cause. After disposal into the tank, irregularities in the waste bed may have formed. The shifting of the waste bed over time as a result of gravitational compression is potentially responsible for the sudden drop in waste level. However, this rationale does not adequately explain the observed increase in tank level without involving another agent (i.e. a slow tank intrusion). Also, further slumping of the waste was not observed in the recent in-tank photographs, and the waste surface was observed to be reasonably uniform.

- Other Possibilities or Combinations of the Preceding Agents

There may be more than one mechanism involved in the observed behavior, such as a long-term slow intrusion coupled with a subsequent upset and relatively rapid loss of material, especially of liquid, because the matrix appears to be quite viscous. There are a limited number of measurements taken and their location is fixed; therefore, local irregularities may be exacerbated. In addition, measurement error and bias also are significant when considering the magnitude of the drop in comparison with the error band.

6.3 TWRs PROGRAM ELEMENT CHARACTERIZATION SYNOPSIS

This section provides selected results obtained from core sampling for some of the most pertinent analytes for the various TWRs program elements, including vitrification, Retrieval, Pretreatment, and Waste Tank Safety. Analytes of interest will be reported on a level of resolution commensurate with the available data and program direction. Watch-list tanks will have segment or subsegment level analyses reported, while non-watch-list tanks are analyzed on a core composite basis. Analytes of interest to multiple programs generally will be reported only in one section. Further detail can be found in the body of the report or in the data packages.

6.3.1 Retrieval Program Data Summary: Physical Properties

A major objective of the characterization program is to measure the physical properties of the waste to support waste retrieval technology development. The analytical methods to determine the physical properties of the waste as it actually exists in the tank require 50 to 100 g of unhomogenized sample. In some cases, the limited amount of sample recovered constrains the number of analyses that can be performed. At the time of the sampling and analysis of tank 241-T-111 waste, no data quality objective existed to define the scope of the analyses. However, several analytes that specifically relate to physical properties were determined to be of interest to the program and are summarized here. The physical characteristics of tank waste are required to develop design criteria for waste retrieval equipment, provide a basis for simulated waste development, and to provide a basis for validation of equipment testing using design criteria and simulated waste. Selected rheological and physical properties are presented in Table 6-1. Further information regarding these analytes can be found in Section 5.3.

Table 6-1. Retrieval Program.

Analyte	Data range
Specific gravity (g/mL)	
--solids	1.19 - 1.28
--liquids (grab sample)	1.036
Shear strength	5,000 \pm 2,300 dynes/cm ²
Viscosity (mPa•s)	
--1:1 dilution @ 29 °C	Less than 2 mPa•s (cP)
Settled solids (Vol %)	100%
Weight % solids	22.4 to 29.3
Weight % undissolved solids	19.0 to 25.4
Particle size (μ m)	
--number distribution	85% < 2 μ m
--volume distribution	70% < 24 μ m

6.3.2 Final Disposal Program Data Summary

Bulk Constituent Concentrations for Pretreatment

Programmatic decisions pertaining to the design of pretreatment and final disposal systems shall be based upon the average characteristics of the tank waste. Therefore, the majority of the laboratory analyses shall be conducted on representative core composites. However, as noted in other documentation (Bell 1993), segment, subsegment, and additional analyses will be performed when directed. The constituent concentrations and inventories shall be calculated by either treating the core samples as random samples and averaging the results, or by using a spatial model. The calculated values will include an estimated total quantity of each selected analyte and its corresponding confidence interval (CI) based upon analytical and sampling variability. Again, no data quality objective existed to define the scope of the analyses at the time tank 241-T-111 was sampled and analyzed. However, several analytes relating specifically to the most significant chemical and radiological contributors and their solubility properties were determined to be of interest to the program and are summarized here. Chemical analytes of interest are presented in Table 6-2. Trace analytes and more comprehensive chemical and radiological characterization information can be found in Section 5.

Low-level and High-Level Vitrification Program

The final disposal option for Hanford-Site wastes has been determined to be vitrification after partitioning into low-level and high-level fractions. This program has characterization needs in addition to those described for core sampling. The vitrification process will be performed after the solids have been pretreated. Therefore, the core sample information will provide preliminary bounding design conditions for the vitrification plant. Further characterization for technology development and regulatory compliance will be necessary on the pretreated waste that will be fed to the vitrification plant. Although the data requirements for this option are not formally defined, the analytical requirements for the previous Hanford Waste Vitrification program generally are applicable and are identified in the *Hanford Waste Vitrification Plant Feed Characterization Requirements*, Revision 4 (Wagner 1992). These requirements are quite similar to the pretreatment program requirements, and therefore are presented together in this section (see Table 6-2). For more specific information on a particular analyte not given in this table, consult the data package (McKinney, et al. 1993) or the appropriate table in Section 5.

The analytical program for vitrification not only entails determining if a waste type is suitable for disposal as glass, but also includes determining the physical and chemical characteristics of the glass for process-control purposes and to ensure regulatory compliance. Sampling and analysis plans will be developed on an individual basis for each tank or process batch. The characterization needs for these efforts include analyses for metals, water-soluble anions, radionuclides, semi-volatile organics, and rheological and physical testing for both the feed and vitrified product.

Table 6-2. Concentrations and Solubility of Principal Waste Components.

Analyte	Tank average concentration water prep: ($\mu\text{g/g}$)	Tank average concentration fusion prep: ($\mu\text{g/g}$)	% Water soluble
Calcium	61.8	2,420	2.55
Chromium	218.3	1,980	11.03
Iron	127.7	18,500	0.69
Aluminum	10.9	570	1.91
Sodium	33,000	37,000	89.19
Bismuth	201.8	26,000	0.78
Lanthanum	11.0	4,220	0.26
Silicon	571.8	5,670	10.08
Uranium	No measurement	3,550	---
Zirconium	0.8	4.0	20.00
Phosphate	15,600 (IC)	32,300	48.29
Sulfate	3,550 (IC)	3,680	96.47
Nitrate	41,300	Not Applicable	---
Fluoride	2,300	Not Applicable	---
TOC	3,120	Not Applicable	---
Radionuclides	($\mu\text{Ci/g}$)	($\mu\text{Ci/g}$)	
^{90}Sr	0.00097 (Grab)	5.41	0.018
^{137}Cs	0.087 (Grab)	0.166	52.33

Tank 241-T-111 presently is not scheduled as an early feed for pretreatment and vitrification. The following characterization objectives need to be addressed in a data quality objective supporting the design of retrieval, pretreatment, and final disposal systems for early feed tanks.

- Provide extensive characterization of the chemical and radiological contents of the waste (solids and supernate) as it currently exists in the tanks to evaluate how it can be processed and to verify if the composition variability study envelope coverage for key analytes is adequate.

- Estimate the waste fraction that will remain after sludge wash pretreatment and estimate the feeds for the low-level and high-level streams for vitrification.
- Simulate sludge washing pretreatment on the waste material. This will provide a detailed understanding of the sludge wash process and obtain empirical data on soluble species removal.
- Determine the physical and rheological properties of the waste before and after simulated sludge washing to support the design of a waste retrieval system.
- Provide a supply of sludge washed material to be used as feed material for a laboratory scale vitrification.
- Satisfy the general characterization requirements for physical, chemical, and radiological analytes.

6.3.3 Waste Tank Safety Program Characterization Data Summary

Safety Screening

The tank safety screening data quality objective will be used to classify 149 SSTs and 28 double-shell tanks that contain high-level radioactive waste into specific safety categories for issues dealing with the presence of ferrocyanide, organics, flammable gases, and criticality (Babad and Redus 1994). The analytes used to make this classification are fuel energy value, total alpha concentration, weight percent moisture, and gas composition. The following table presents the analytes of concern, the criterion for classification, and the analytical result from the tank, where available. Further information on the tank contents are presented in subsequent sections. Because of the exothermic response of the waste material from the upper portions of tank 241-T-111, it has been placed on the Organic Watch List, and further studies trying to resolve the nature of the observed reaction are continuing. Table 6-3 provides a comparison of the tank values with the safety screening criteria.

Table 6-3. Tank 241-T-111 Comparison to Safety Screening Criteria.

Analyte	Safety issue/criteria	Tank result
Fuel energy value (cal/dry g)	Organics, ferrocyanide, flammable gas; -125 cal/dry g	Greater than -215 cal/dry g
Total alpha concentration	Criticality; 1 g ²³⁹ Pu/L	0.0053 g/L
Percent moisture	Organics, ferrocyanide, flammable gas; 17 wt %	76 wt %
Gas composition	25% lower flammability limit	NA

NA = Not available

Criticality Safety

The criticality safety program has indicated that plutonium and uranium isotopic analyses on each core composite and the bottom six inches of each core is required to alleviate the concern for the potential of tank criticality. Therefore, upon extruding the last segment in a core, the waste to be tested shall be homogenized before a small aliquot is taken and analyzed for plutonium and uranium isotopic analyses by mass spectroscopy. The analyses will indicate whether the fissile species have settled in a concentrated layer at the bottom of a tank. However, this analytical criterion was established after the sampling and analysis of tank 241-T-111 waste. Tables 6-4 and 6-5 present a summary of the core composite data for uranium and plutonium concentration. After reviewing the data, the isotopic analyses from both cores 31 and 33 were found to be very consistent with regard to composition, and the total alpha content indicated from the core 33, segment 9 homogenization data is extremely low (0.262 $\mu\text{Ci/g}$). Therefore further re-analysis and isotopic resolution of the core sample material from the lower portion of cores 31 and 33, segment 9 is not warranted. For this tank to exceed established operating limits for fissile material in the tank farms, a concentration of 1.58 $\mu\text{Ci/g}$ $^{239/240}\text{Pu}$ was calculated as a threshold limit value (see Appendix C).

Table 6-4. Core Composite Uranium.

Core No.	U_{FL} (222-S) ($\mu\text{g/g}$)	U_{FL} (325) ($\mu\text{g/g}$)	^{238}U mass percent	^{235}U mass percent
Core 31, composite 1	2,180	4,000	99.3074	0.6755
Core 31, composite 2	3,880	5,200	99.3098	0.6761
Core 33, composite 1	3,180	4,500	99.3125	0.6761
Core 33, composite 2	1,950	3,500	99.3161	0.6717

FL = Uranium measurement by laser fluorimetry.

Table 6-5. Plutonium Concentration and Isotopic Distribution

Core number	Total Pu α (222-S) ($\mu\text{Ci/g}$)	Total Pu α (325) ($\mu\text{Ci/g}$)	^{238}Pu mass percent	^{239}Pu mass percent	^{240}Pu mass percent	^{241}Pu mass percent	^{242}Pu mass percent
Core 31 C1	0.138	0.628	0.005	96.7199	3.2109	0.0352	0.0151
Core 31 C2	0.136	0.565	0.0105	96.6351	3.2834	0.0496	0.0215
Core 33 C1	0.134	0.319	0.004	96.7540	3.1046	0.1071	0.0683
Core 33 C2	0.147	0.368	0.0105	96.5499	3.3436	0.0621	0.0337

Organic Tanks

The following characterization objectives support resolution of this unreviewed safety question and safety issue and support retrieval, pretreatment, and final disposal systems design. Table 6-6 provides a comparison of the tank core composite values with the Organic Data Quality Objective Criteria.

- Determine the overall waste energetics and properties governing waste reactivity behavior in the tanks.
- Determine the spatial distribution of ^{137}Cs and ^{90}Sr .
- Determine the concentration of TOC and the speciation of organics present in the waste.
- Satisfy the general characterization requirements for physical, chemical, and radiological analytes.

Table 6-6. Data Quality Objective Decision Limits for Organic Tanks. (Babad 1994)

Analyte	Decision threshold	Tank result
TOC (Dry wt%)	> 5 wt% (dry basis)	1.3 wt% (tank) 4.1 wt% (Core 33, Seg. 2)
Moisture content (wt%)	< 17 wt%	76 wt%
Presence of organic layer	Yes/No	No
Tank temperature (°C)	90 °C	16 °C
Total fuel content	-125 cal/dry g	In excess of -215 cal/dry g

Further analysis and secondary analytes for measurement were found to be unwarranted for the tank composites; however, the first two segments of core 31 and three segments of core 33 will undergo additional study. The results from this series of assays will be incorporated into the revision of this characterization report as they become available.

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7.0 QUANTITATIVE/STATISTICAL INTERPRETATION OF THE DATA

This section contains the results of the statistical analysis of data from two core samples obtained from tank 241-T-111. Section 7.1 contains a description of the core-sample data used in the statistical analyses and some general observations regarding the data. Section 7.2 contains mean concentration estimates, and the associated 95 percent CIs, for each of the analytes in tank 241-T-111 waste. Section 7.3 contains estimates of the spatial variability (variability between cores), and estimates of the analytical error from the core composite data in tank 241-T-111. Further information can be found in Jensen (1994).

Two types of analytical error were estimated from the core composite data: variability between composite samples within the same core and variability between the primary and duplicate analyses within each core composite sample. Estimates of the analytical measurement error were used to test the significance of the spatial and compositing variability. Spatial variability was significant (i.e. substantially greater than zero at the 0.05 significance level) in 40 out of 79 analytes in the tank. The compositing variance was significant for 38 out of the 79 analytes.

7.1 APPROACH

Cores 31 and 33 were the two valid core samples taken from tank 241-T-111. The segment recoveries for each core were given previously in Section 4. Two core composite samples were made for each core from the homogenized solid segment waste. Primary and duplicate results were obtained from each core composite.

The ICP acid digestion, ICP fusion dissolution, ICP water leach, IC water leach analyses, selected radiochemistry, and other GEA were performed on all composite core samples. These were the analytical results used in the statistical treatment of the data. In the tables in Appendix B, the data are identified by the analysis method, the type of dissolution, and analyte; e.g., the notation ICP.a.Al refers to aluminum, acid digestion, and an ICP analysis. The core composite sample results used also are contained in Appendix B, together with ratios of the mean of each sample and duplicate divided by the detection limit for that pair. The data package for tank 241-T-111 (McKinney et al. 1993) contains a complete report of the sample results along with the laboratory quality control data. The core composite data for each analyte are illustrated in Appendix B.

Statistics were calculated for analytes with concentrations greater than 10 times their detection limits (DL). Personnel within the TWRS Information Management Systems have identified a list of analytes that have exception to this rule. Table 7-1 lists the analytes specified. Statistics were calculated for the analytes from this list if the concentrations were greater than three times their DL. For a number of analytes, the concentrations of some samples were greater than a particular limit (3 or 10 times the DL), while the other samples were less than that limit. In these cases the statistics were calculated using all of the data

whether it was above or below the particular limit (3 or 10 DL). The above rules do not apply to alpha or beta/gamma counting methods. The ratios (mean/DL) reported in Appendix B are provided to show how large the analyte concentrations are relative to the DL.

Table 7-1. Special Analyte List.

Aluminum	Nitrate
Bismuth	Nitrite
Calcium	Phosphate
Chromium	Carbonate
Iron	Fluoride
Silicon	Chloride
Sodium	TOC
Zirconium	Cyanide

A close examination of the figures in Appendix B reveals several outlier data points in the core composite data:

ICP.a.Co: The primary result for core 31, composite 2 of 11.7 ug/g is over three times the duplicate result. The other results for ICP.a.Co all fall in the range 2.7 ug/g to 3.8 ug/g. The detection limit for ICP.a.Co is 0.8 ug/g.

ICP.a.Cu: The duplicate result for core 31, composite 2 of 127 ug/g is about four times the primary result of 31.7 ug/g. The detection limit for ICP.a.Cu is 0.4 ug/g.

NO₂: Both the primary and duplicate results from composite 2 of core 31 are about half the results for core 31, composite 1. The average of the results for core 31, composite one is 952 ug/g; the average for composite two is 525 ug/g. The detection limit for NO₂ by water digestion spectrophotometric analysis is 50 ug/g.

7.2 MEAN CONCENTRATION ESTIMATES

One of the tasks outlined as part of the waste characterization effort (Bell 1993, Winters et al. 1990a, Winters et al. 1990b), is to estimate the constituent inventories in the waste. The inventories are estimated by computing mean concentrations and 95 percent CIs on the mean concentrations for each constituent. The estimate of the inventory and CI on the inventory of an analyte in the tank are equal to the corresponding mean concentration estimates and CI multiplied by the volume of waste in the tank.

7.2.1 Statistical Methods

The concentration estimates are given in the form of 95 percent CIs on the mean concentration. It is assumed that each sample and its duplicate are analyzed independently of one another. The two analytical results are used to estimate the analytical measurement error. Because of the hierarchical structure of the data, the analytical measurement error alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variance and the spatial variance is the appropriate variance of the mean for the CIs. Appendix B contains a description of the statistical model and formulas used to calculate estimates of the mean, variance of the mean, and the CI on the mean.

7.2.2 Statistical Results

Table 7-2 contains the summary statistics by analyte for ICP acid digestion, ICP water leach, ICP fusion dissolution, IC, and selected radiochemical and physical analyses. The summary statistics are as follows:

\bar{y} mean of the concentration data

$\hat{\sigma}^2(\bar{y})$ estimated variance of \bar{y}

df degrees of freedom

95 % LL lower limit to the 95 percent CI on the mean

95 % UL upper limit to the 95 percent CI on the mean.

For some analytes the lower confidence limit (95 percent LL) was negative. Because concentrations are greater than or equal to zero, any negative 95 percent LL values were set equal to zero.

The CIs in Table 7-2 are wide relative to the range of the data. The CIs are wide because only two cores were used to estimate the spatial variability. A minimum of two core samples is needed to estimate a tank's spatial variability.

Table 7-2. Concentration Estimate Statistics. (4 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\hat{\sigma}^2(\bar{y})$	df	95% LL	95% UL
ICP.a.Ag	1.26E+02	7.86E+03	1	0.00	1.25E+03
ICP.a.Al	5.41E+02	1.06E+04	1	0.00	1.85E+03
ICP.a.B	2.80E+01	7.56E+00	1	0.00	6.30E+01
ICP.a.Ba	6.90E+01	6.46E+01	1	0.00	1.71E+02
ICP.a.Bi	2.59E+04	6.38E+06	1	0.00	5.80E+04
ICP.a.Ca	1.88E+03	2.12E+05	1	0.00	7.72E+03
ICP.a.Cd★	5.80E+00	3.03E+00	1	0.00	2.79E+01
ICP.a.Co●	4.30E+00	1.63E+00	1	0.00	2.05E+01
ICP.a.Cr★	1.98E+03	1.63E+04	1	3.57E+02	3.60E+03
ICP.a.Cu	3.35E+01	3.53E+02	1	0.00	2.72E+02
ICP.a.Fe	1.85E+04	1.21E+06	1	4.55E+03	3.25E+04
ICP.a.K	1.14E+03	2.24E+03	1	5.34E+02	1.74E+03
ICP.a.La	4.22E+03	3.00E+05	1	0.00	1.12E+04
ICP.a.Mg	3.77E+02	6.36E+03	1	0.00	1.39E+03
ICP.a.Mn	6.33E+03	2.68E+04	1	4.25E+03	8.41E+03
ICP.a.Na	3.69E+04	1.56E+06	1	2.10E+04	5.27E+04
ICP.a.Ni	1.32E+02	5.12E+02	1	0.00	4.19E+02
ICP.a.P	1.03E+04	1.21E+05	1	5.90E+03	1.47E+04
ICP.a.Pb	3.47E+02	2.64E+04	1	0.00	2.41E+03
ICP.a.S	1.21E+03	1.06E+03	1	8.00E+02	1.63E+03
ICP.a.Si	4.69E+02	9.17E+02	1	8.40E+01	8.54E+02
ICP.a.Sr	3.00E+02	3.75E+02	1	5.39E+01	5.46E+02
ICP.a.Ti	1.95E+01	1.39E+02	1	0.00	1.69E+02
ICP.a.V	1.45E+01	6.58E+00	1	0.00	4.71E+01
ICP.a.Zn	6.50E+01	6.46E+02	1	0.00	3.88E+02

Table 7-2. Concentration Estimate Statistics. (4 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\hat{\sigma}^2(\bar{y})$	df	95 % LL	95 % UL
ICP.f.Ag	1.28E+02	8.05E+03	1	0.00	1.27E+03
ICP.f.Al	5.70E+02	9.70E+03	1	0.00	1.82E+03
ICP.f.Ba	6.46E+01	2.45E+01	1	1.73E+00	1.28E+02
ICP.f.Bi	2.36E+04	9.08E+06	1	0.00	6.18E+04
ICP.f.Ca★	2.42E+03	8.27E+04	1	0.00	6.07E+03
ICP.f.Cd★	8.12E+00	1.76E+00	1	0.00	2.50E+01
ICP.f.Cr	1.80E+03	1.56E+03	1	1.30E+03	2.30E+03
ICP.f.Cu	2.93E+01	3.56E+01	1	0.00	1.05E+02
ICP.f.Fe	1.80E+04	4.05E+06	1	0.00	4.36E+04
ICP.f.La	4.11E+03	3.08E+05	1	0.00	1.12E+04
ICP.f.Mg	3.55E+02	7.31E+03	1	0.00	1.44E+03
ICP.f.Mn	6.28E+03	1.88E+04	1	4.54E+03	8.02E+03
ICP.f.Na	3.70E+04	6.00E+06	1	5.82E+03	6.81E+04
ICP.f.Ni	8.14E+03	6.41E+06	1	0.00	4.03E+04
ICP.f.P	1.04E+04	8.42E+05	1	0.00	2.21E+04
ICP.f.Pb★	3.65E+02	9.38E+03	1	0.00	1.60E+03
ICP.f.S	1.23E+03	1.13E+04	1	0.00	2.58E+03
ICP.f.Si	5.67E+03	5.41E+04	1	2.71E+03	8.62E+03
ICP.f.Sr	2.98E+02	6.24E+01	1	1.97E+02	3.98E+02
ICP.f.Ti	4.79E+01	6.09E+02	1	0.00	3.62E+02
ICP.f.Zn★	1.06E+02	7.17E+00	1	7.22E+01	1.40E+02
ICP.w.Al●	1.09E+01	5.75E+00	1	0.00	4.14E+01
ICP.w.Bi	2.02E+02	2.40E+03	1	0.00	8.24E+02
ICP.w.Ca★	6.16E+01	3.32E+01	1	0.00	1.35E+02
ICP.w.Cr	2.18E+02	2.45E+01	1	1.55E+02	2.81E+02
ICP.w.Fe	1.28E+02	3.11E+02	1	0.00	3.52E+02

Table 7-2. Concentration Estimate Statistics. (4 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\hat{\sigma}^2(\bar{y})$	df	95% LL	95% UL
ICP.w.K	7.19E+02	1.54E+03	1	2.21E+02	1.22E+03
ICP.w.La★	1.10E+01	1.42E+01	1	0.00	5.89E+01
ICP.w.Mg★	3.64E+00	5.45E-02	1	6.75E-01	6.61E+00
ICP.w.Mn	2.47E+01	2.36E+01	1	0.00	8.65E+01
ICP.w.Na	3.30E+04	2.44E+06	1	1.31E+04	5.28E+04
ICP.w.P	5.68E+03	3.24E+04	1	3.39E+03	7.97E+03
ICP.w.S	1.15E+03	2.38E+03	1	5.29E+02	1.77E+03
ICP.w.Si	5.72E+02	5.35E+03	1	0.00	1.50E+03
IC.w.Cl ⁻	4.50E+02	1.11E+03	1	2.56E+01	8.74E+02
IC.w.F ⁻	2.30E+03	6.46E+05	1	0.00	1.25E+04
IC.w.NO ₂ ★	8.97E+02	2.10E+04	1	0.00	2.74E+03
IC.w.NO ₃ ⁻	4.12E+04	7.77E+06	1	5.82E+03	7.67E+04
IC.w.PO ₄ ³⁻	1.55E+04	1.53E+06	1	0.00	3.13E+04
IC.w.SO ₄ ²⁻	3.54E+03	2.85E+04	1	1.40E+03	5.69E+03
GEA.Am-241★	4.24E-02	2.61E-06	1	2.19E-02	6.29E-02
GEA.Co-60	3.64E-04	2.68E-10	1	1.56E-04	5.72E-04
GEA.Cs-137	1.66E-01	3.35E-03	1	0.00	9.02E-01
Gross.alpha	3.73E-01	1.96E-04	1	1.95E-01	5.51E-01
Gross.beta	1.51E+01	3.48E+01	1	0.00	9.00E+01
TGA.Percent.H ₂ O	7.65E+01	2.23E+01	1	1.64E+01	1.37E+02
NO ₂ ⁻	7.93E+02	8.76E+03	1	0.00	1.98E+03
Percent.H ₂ O	7.60E+01	5.81E-01	1	6.63E+01	8.57E+01
Pu-239/240	1.39E-01	9.19E-06	1	1.00E-01	1.77E-01
Sr-90	5.41E+00	3.53E+00	1	0.00	2.93E+01
TOC★	3.12E+03	3.83E+05	1	0.00	1.10E+04
Tc-99●	7.92E-03	8.90E-06	1	0.00	4.58E-02

Table 7-2. Concentration Estimate Statistics. (4 pages)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$\hat{\sigma}^2(\bar{y})$	df	95% LL	95% UL
U★	2.79E+03	2.01E+05	1	0.00	8.50E+03
pH	9.98E+00	7.79E-03	1	8.86E+00	1.11E+01

●: Analytes with a portion of the data below 3 times the DL.

★: Analytes with a portion of the data below 10 times the DL.

7.3 COMPARISON OF THE VARIANCE COMPONENT ESTIMATES

Using the hierarchical structure of the core composite data, estimates of the between-core spatial variability, the compositing variability, and the analytical-measurement variability can be obtained. The spatial variance is a measure of the variability between cores. The compositing variance measures the variability between composite samples within the same core. The analytical-measurement variance is a measure of the difference between the analytical results from the sample and duplicate samples. This variance includes, among other things, the sample handling error and the chemical analysis error.

The estimate of the variance of the mean is a linear function of the spatial, compositing, and analytical-measurement variances. To help evaluate the magnitude of these three variance components, estimates of each variance component are given.

7.3.1 Statistical Methods

Estimates of the spatial variance ($\hat{\sigma}^2(S)$), compositing variance ($\hat{\sigma}^2(C)$), and analytical-measurement variance ($\hat{\sigma}^2(A)$), were obtained for each analyte using restricted maximum likelihood estimation methods. Restricted maximum likelihood estimation is discussed by Harville (1977).

To test the significance of the variance components, an analysis of variance (ANOVA) was calculated using the hierarchical statistical model described in Appendix B. The mean square error terms in the ANOVA table were used to perform an F-test on the spatial variability and the composite variability. The p-values given in Table 7-3 were derived from the results of these tests.

7.3.2 Statistical Results

The restricted maximum likelihood estimates of each component of variability along with the p-values (significance level) from the F-tests also are given in Table 7-3. P-values less than 0.05 indicate that $\sigma^2(S)$ or $\sigma^2(C)$ is significantly different from zero at the 0.05 significance level.

The p-values from the tests on $\sigma^2(S)$ were less than 0.05 for 40 out of the 79 analytes in tank 241-T-111 waste. Thus, for these 40 cases, differences between the results in the two cores were statistically significant^{**}. The p-values from the tests on $\sigma^2(C)$ were less than 0.05 for 38 out of the 79 analytes in tank 241-T-111 waste. This indicates that, relative to the analytical error, differences between composite samples were significantly greater than zero in 38 cases. Conversely, for 41 out of 79 cases, differences between composite samples were not statistically significant. The number of analytes (and the amount that they contribute to the waste) for which $\sigma^2(S)$ and $\sigma^2(C)$ were statistically significant further suggests that the waste is heterogeneous.

7.4 MASS BALANCES

A method to help ensure the data are consistent and reasonable is to perform a mass and charge balance on the core composite sample data. This activity is a rough quality control check and provides insight to some of the properties of the matrix. To do this, the assumption in performing the mass balance is that the anions, cations, and water are all associated in some manner, but the exact chemistry of the association is not considered. Analytes contributing less than 0.2 weight percent, generally trace ICP analytes, AA-analytes, and radionuclides, are considered negligible in this assessment. The assays that will contribute analytes to the mass balance are ICP acid or fusion (whichever gives higher quantitation), IC, TOC, and the gravimetric weight-percent water measurement.

Without considering the physical and chemical properties of the waste matrix and the context of the process history, the mass balances produced from these assays may be biased low. However, this bias is expected because it is known that there are analytes present that were not measured in the analysis of the samples. The IC anions only measure the water-soluble components; there is a substantial insoluble residue that must contain additional anions. Bias may be impacted substantially by chemical form, accountability, and variability in oxide or hydroxide content. Assumptions regarding the chemical combination of some of the analytes will be made and inserted into the mass/charge balance, presented in Table 7-4. Generally, this consists of assuming that some analytes are precipitated as an oxide or hydroxide, and that the shortfall indicated from the charge balance in microequivalent [$\mu\text{equ.}$] is present as one of those two analytes.

^{**}Statistically significant for the purposes of this analysis means substantially greater than zero at the 0.05 level of significance.

Table 7-3. Variance Components Estimates. (3 pages)

Analyte	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.a.Ag	1.56E+04	0.001	1.94E+02	0.000	3.16E+00
ICP.a.Al	1.89E+04	0.055	4.79E+03	0.000	1.06E+01
ICP.a.B	1.25E+01	0.093	9.28E-01	0.387	8.59E+00
ICP.a.Ba	6.91E+01	0.263	1.20E+02	0.000	3.54E-01
ICP.a.Bi	1.27E+07	0.000	6.25E+03	0.360	3.75E+04
ICP.a.Ca	4.10E+05	0.010	2.17E+04	0.074	9.95E+03
ICP.a.Cd*	5.96E+00	0.004	1.90E-01	0.039	5.34E-02
ICP.a.Co*	8.92E-01	0.376	8.08E-01	0.389	7.84E+00
ICP.a.Cr	3.17E+04	0.007	1.36E+03	0.081	6.75E+02
ICP.a.Cu	3.39E+02	0.290	1.67E+02	0.369	1.14E+03
ICP.a.Fe	2.33E+06	0.013	1.55E+05	0.063	6.25E+04
ICP.a.K	2.45E-24	0.911	8.86E+03	0.000	1.75E+02
ICP.a.La	5.91E+05	0.003	1.63E+04	0.019	2.88E+03
ICP.a.Mg	1.25E+04	0.005	5.18E+02	0.001	1.58E+01
ICP.a.Mn	2.97E+04	0.254	4.29E+04	0.030	1.00E+04
ICP.a.Na	2.76E+06	0.057	6.73E+05	0.016	1.05E+05
ICP.a.Ni	1.02E+03	0.000	6.13E+00	0.091	3.38E+00
ICP.a.P	2.37E-22	0.608	4.77E+05	0.000	1.25E+04
ICP.a.Pb	5.20E+04	0.003	1.41E+03	0.000	1.91E+01
ICP.a.S	1.11E+03	0.268	1.94E+03	0.003	1.25E+02
ICP.a.Si	2.57E-31	0.946	7.61E-14	0.418	7.34E+03
ICP.a.Sr	6.38E+02	0.079	2.18E+02	0.004	1.54E+01
ICP.a.Ti	2.75E+02	0.001	4.69E+00	0.000	7.83E-02
ICP.a.V	1.83E+00	0.423	2.25E+01	0.000	2.30E-01
ICP.a.Zn	1.22E+03	0.021	1.35E+02	0.008	1.42E+01
ICP.f.Ag	1.61E+04	0.000	6.23E+00	0.295	1.83E+01
ICP.f.Al	1.90E+04	0.005	6.78E+02	0.030	1.57E+02

Table 7-3. Variance Components Estimates. (3 pages)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
ICP.f.Ba	3.99E+01	0.102	1.71E+01	0.012	2.19E+00
ICP.f.Bi	1.81E+07	0.001	2.88E+04	0.392	2.94E+05
ICP.f.Ca★	1.51E+05	0.008	2.07E-19	0.804	5.86E+04
ICP.f.Cd★	2.17E+00	0.126	1.21E-20	0.649	5.38E+00
ICP.f.Cr	3.72E-15	0.994	5.63E+03	0.013	1.21E+03
ICP.f.Cu★	6.97E+01	0.005	2.00E+00	0.119	1.43E+00
ICP.f.Fe	8.00E+06	0.003	1.68E+05	0.079	8.13E+04
ICP.f.La	5.96E+05	0.010	3.82E+04	0.012	4.98E+03
ICP.f.Mg	1.46E+04	0.000	5.15E-44	0.913	1.60E+02
ICP.f.Mn	1.66E-11	0.553	7.02E+04	0.011	9.70E+03
ICP.f.Na	1.17E+07	0.007	5.43E+05	0.044	1.65E+05
ICP.f.Ni	1.17E+07	0.033	4.18E-12	0.498	4.39E+06
ICP.f.P	1.57E+06	0.028	1.95E+05	0.049	6.45E+04
ICP.f.Pb★	1.85E+04	0.003	4.35E+02	0.030	1.02E+02
ICP.f.S	2.15E+04	0.017	2.00E+03	0.008	2.13E+02
ICP.f.Si	1.06E+05	0.005	1.00E+03	0.369	6.83E+03
ICP.f.Sr	6.12E-16	0.661	1.15E+02	0.222	2.69E+02
ICP.f.Ti	1.22E+03	0.000	2.66E-01	0.331	1.12E+00
ICP.f.Zn★	5.69E-23	0.440	1.10E-22	0.839	5.74E+01
ICP.w.Al●	7.35E+00	0.208	6.93E+00	0.061	2.72E+00
ICP.w.Bi	3.90E+03	0.102	1.30E+03	0.123	9.66E+02
ICP.w.Ca★	1.86E-11	0.144	1.69E-22	0.862	2.66E+02
ICP.w.Cr	5.66E-20	0.979	9.52E+01	0.001	5.62E+00
ICP.w.Fe	6.16E+01	0.436	9.05E+02	0.077	4.29E+02
ICP.w.K	2.28E+03	0.148	1.57E+03	0.001	4.73E+01
ICP.w.La★	2.71E+01	0.015	1.34E+00	0.226	2.23E+00
ICP.w.Mg★	3.04E-21	0.597	1.61E-01	0.098	1.14E-01

Table 7-3. Variance Components Estimates. (3 pages)

Analyte	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.w.Mn	2.51E+01	0.266	3.19E+01	0.130	2.51E+01
ICP.w.Na	4.53E+06	0.030	6.84E+05	0.002	2.88E+04
ICP.w.P	3.98E+04	0.222	3.94E+04	0.089	2.13E+04
ICP.w.S	3.95E+03	0.091	1.50E+03	0.014	2.13E+02
ICP.w.Si	8.53E+03	0.113	1.98E+03	0.272	4.72E+03
IC.w.Cl	1.99E+03	0.043	1.15E-10	0.509	9.66E+02
IC.w.F ⁻	1.28E+06	0.001	1.37E+04	0.102	8.44E+03
IC.w.NO ₂ ★	3.99E+04	0.015	5.22E-16	0.510	8.64E+03
IC.w.NO ₃ ⁻	1.42E+07	0.038	2.46E+06	0.013	3.44E+05
IC.w.PO ₄ ³⁻	2.59E+06	0.082	8.54E+05	0.030	1.99E+05
IC.w.SO ₄ ²⁻	5.27E+04	0.032	7.04E+03	0.071	3.14E+03
GEA.Am-241★	2.99E-32	0.786	8.13E-06	0.062	4.59E-06
GEA.Co-60	3.58E-10	0.046	5.14E-36	0.813	7.12E-10
GEA.Cs-137	6.61E-03	0.003	1.87E-04	0.000	1.88E-06
Gross.alpha	3.42E-04	0.065	7.74E-05	0.098	4.58E-05
Gross.beta	6.93E+01	0.000	3.14E-01	0.026	6.62E-02
TGA.%H ₂ O	3.17E+01	0.008	3.78E-33	0.935	5.18E+01
NO ₂	1.92E-14	0.789	3.46E+04	0.000	9.50E+02
Percent H ₂ O	8.99E-01	0.128	2.55E-01	0.259	5.46E-01
Pu-239/240	2.49E-28	0.736	2.01E-05	0.171	3.33E-05
Sr-90	7.04E+00	0.000	1.79E-02	0.209	2.61E-02
TOC★	6.10E+05	0.113	2.98E+05	0.006	2.72E+04
Tc-99●	1.76E-05	0.001	2.63E-07	0.027	5.75E-08
U★	6.24E-06	0.831	7.91E+05	0.001	2.99E+04
pH	7.11E-28	0.601	3.06E-02	0.001	1.19E-03

●: Analytes with a portion of the data below 3 times the DL.

★: Analytes with a portion of the data below 10 times the DL.

Table 7-4. Core 31 and Core 33 Mass and Charge Balance. (2 pages)

Analyte	Core 31 average concentration ($\mu\text{g/g}$)	Core 31 charge ($\mu\text{equ/g}$)	Core 33 average Concentration ($\mu\text{g/g}$)	Core 33 charge ($\mu\text{equ/g}$)	Core 31/33 RPD
Ca^{+2}	2,710	135.50	2,140	107.00	23.51
Cr^{+3}	1,850	106.73	2,100	121.15	-12.66
Fe^{+2}	20,100	717.86	16,000	571.43	22.71
Mn^{+4}	6,160	448.00	6,400	465.45	-3.82
Na^{+}	39,400	1,713.04	34,600	1,504.35	12.97
Bi^{+3}	23,500	337.32	28,500	409.09	-19.23
La^{+3}	3,670	79.21	4,770	102.95	-26.07
Si^{+4}	5,900	842.86	5,440	777.14	8.11
U^{+6}	3,820	96.30	3,280	82.69	15.21
PO_4^{3-}	30,100	-950.53	31,700	-1001.05	-5.18
SO_4^{2-}	3,650	-76.04	3,460	-72.08	5.34
NO_3^{-}	44,100	-711.29	38,500	-620.97	13.56
F^{-}	3,110	-163.68	1,500	-78.95	69.85
TOC	3,740	-85.00	2,500	-56.82	39.74
Anion reconciliation					
O_2^{4-}	17,116	-2,139.50	16,926	-2,115.75	
OH^{-}	5,965	-350.88	3,325	-195.59	
Water content					
H_2O	735,000	0	790,000	0	-7.21
H_2O Calc.	785,109	0	798,859	0	-1.74

Table 7-4. Core 31 and Core 33 Mass and Charge Balance. (2 pages)

Analyte	Core 31 average concentration ($\mu\text{g/g}$)	Core 31 charge ($\mu\text{equ/g}$)	Core 33 average Concentration ($\mu\text{g/g}$)	Core 33 charge ($\mu\text{equ/g}$)	Core 31/33 RPD
Total	949,891	-0.10	991,141	0.05	
Percent difference*	-5.01		-0.89		

O_2^{4-} : Represents the overall mass and charge of oxygen added to manganese, uranium, silicon, TOC, and chromium as part of the assumptions given in Section 7.4. This notation is not meant to imply actual chemical form.

OH: Represents hydroxide amount calculated to reconcile charge balance

NOTE: Neither of these analytes (oxygen or OH) are analytically determined.

$$\text{Relative Percent Difference (RPD)} = \frac{\text{Core 31 value} - \text{Core 33 value}}{\left(\frac{\text{Core 31 value} + \text{Core 33 value}}{2} \right)} \times 100$$

*Percent difference is determined from a Total of $1.0\text{E}+06 \frac{\mu\text{g}}{\text{g}}$ i.e.,

$$\text{Percent Difference} = \frac{\text{sum of analytical values in a core} - 1,000,000}{1,000,000} \times 100$$

A significant source of error can be reduced by assuming all phosphorous is present as PO_4^{3-} . The water digestion ICP values for phosphorous (converted to PO_4^{3-}) and PO_4^{3-} values from the IC agree well. The ratio of soluble to insoluble phosphorous (taken as phosphate) indicates that it is only about 50 percent soluble. The process history of the tank also indicates that large amounts of phosphate were used to encourage precipitate formation. Therefore, an assumption that the phosphorus determined by ICP in the fusion acid/assay (and converted to phosphate) represents total PO_4^{3-} is not unwarranted. The phosphorus in the ICP fusion assay is converted to PO_4^{3-} and added to the other anions in the charge balance. This step will avoid double counting in the mass and charge balance calculations.

The following other assumptions will be made for the purposes of simplifying the calculations: manganese is assumed present as MnO_2 , uranium is present as $\text{UO}_2(\text{OH})_2$, silicon is present as SiO_2 , TOC as $\text{C}_2\text{O}_4^{2-}$, and chromium as Cr_2O_3 . These forms are not the

only likely speciation of the analytes; however, the waste matrices are too complicated to represent every possible, or even probable compound present.

In the case of these waste materials, the disparity between the gravimetric water measurement and the TGA water content suggests (1) drying of the sample before the gravimetric assay; (2) incomplete drying during the gravimetric test, which biases the results low; or (3) one or more endothermic events occurring in the same temperature range (chemical reactions or phase transitions resulting in the loss of mass). One or more of these factors may be responsible for the observed trend. However, in this case, the analytical results and chemical assumptions that were made with regard to the waste matrix reconcile well.

7.5 SUGGESTED COMPONENTS OF WASTE MATRIX

The actual composition of the waste matrix is quite complex and trace amounts of various compounds probably exist in the tank. However, with some simple assumptions regarding how the anions and cations will combine, a list of the most probable compounds that exist in the waste matrix and contribute significantly to its overall makeup can be developed.

Table 7-5 is a condensed version of a more general chart found on page D-147 in the *Handbook of Chemistry and Physics 64th Ed.* (Weast 1984). It provides solubility data on some of the most common anions and cations. The oxidation state shown in the table for the cations is the most stable. However, precipitates may form for multivalent cations under varying conditions, and so precipitates are reported as likely, if conditions and anions in the assessment of the analyst warrant it.

From the chromatographic data, suspected solubility behavior, and process information, chloride, nitrite, and carbonate will not be significant mass contributors to the waste matrix. Sodium, SO_4^{2-} , and NO_3^- are highly soluble, and thus probably do not contribute much to the insoluble solids. However, they contribute significantly to the overall solids content of the waste (dissolved + insoluble solids). Phosphorous is one of the most prevalent analytes, is approximately 50 percent soluble, and contributes substantially to both the soluble and insoluble solids. No analytical measurement of hydroxide was made for the solids (although there was an OH^- assay of the grab sample), but it is known that in the process history of tank 241-T-111, basic solutions were added routinely to the tank. The following are likely candidates for the insoluble solids:

- Bismuth phosphate, BiPO_4
- Bismuth hydroxide, Bi(OH)_3
- Calcium fluoride, CaF_2
- Bismuth fluoride, BiF_3
- Bismuth trioxide, Bi_2O_3
- Calcium phosphate, $\text{Ca}_3(\text{PO}_4)_2$

Table 7-5. Probable Solids in the Waste Matrix.

	NO ₃	PO ₄ ³⁻	SO ₄ ²⁻	F ⁻	OH ⁻	Si (as SiO ₂)	Oxide 2	CO ₃ ²⁻
Bi ⁺³		PPT		PPT	PPT		PPT	
Ca ⁺²		PPT	PPT	PPT		PPT	PPT	PPT
Cr ⁺³		PPT	PPT	PPT	PPT		PPT	
Fe ⁺³		PPT		PPT	PPT		PPT	PPT
Na ⁺								
La ⁺³			PPT	PPT	PPT		PPT	PPT
Mn ⁺⁴		PPT		PPT	PPT	PPT	PPT	PPT
U ⁺⁶	NL	PPT	PPT	NL	PPT		PPT	

PPT = Precipitate forms.

NL = Precipitate formation not likely under tank conditions.

- Calcium carbonate, CaCO₃
- Calcium oxide, CaO
- Calcium sulfate, CaSO₄
- Chromium(II) fluoride, CrF₂
- Chromium phosphate, CrPO₄•2H₂O
- Chromium dioxide, CrO₂
- Chromium oxide, Cr₂O₃
- Iron(III) fluoride, FeF₃
- Iron(III) hydroxide, Fe(OH)₃
- Iron(III) phosphate, FePO₄
- Lanthanum oxide, La₂O₃
- Manganese difluoride, MnF₂
- Manganese phosphate, MnPO₄•H₂O
- Manganese dioxide, MnO₂
- Manganese(III) hydroxide, MnO(OH)
- Uranyl hydroxide, UO₂(OH)₂
- Calcium chromite, CaCr₂O₄
- Calcium silicate, CaSiO₃
- Calcium hydroxide, Ca(OH)₂
- Chromium(III) fluoride, CrF₃
- Chromium hydroxide, Cr(OH)₃
- Chromium monoxide, CrO
- Iron(II) fluoride, FeF₂
- Iron(II) hydroxide, Fe(OH)₂
- Iron(II) phosphate, Fe₃(PO₄)₂
- Lanthanum hydroxide, La(OH)₃
- Lanthanum fluoride, LaF₃
- Manganese trifluoride, MnF₃
- Manganese hydroxide, Mn(OH)₂
- Manganese oxide, Mn₃O₄
- Uranyl phosphate, UO₂HPO₄•4H₂O
- Uranyl sulfate, 2(UO₂SO₄)•7H₂O.

Insoluble aluminosilicates are suspected of binding the ¹³⁷Cs. ⁹⁰Sr may be held by several possible insoluble ionic compounds. There are many more possible and complex compounds that conceivably could exist in the waste matrix. This list is not meant as authoritative or exhaustive, and the alkaline nature of the media may substantially alter the phase equilibria for some of these materials. However, it does provide a reasonable starting point for any further speciation work.

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8.0 CONCLUSIONS

Analyses of the waste show a very small number of analytes comprising a disproportionate majority of the waste. Water is the single largest analyte, making up over 75 percent of the solids mass. Less than one percent of the total mass of the tank waste is drainable liquid. Calcium, chromium, iron, manganese, sodium, lanthanum, bismuth, and silicon constitute approximately 10.7 percent of the solids mass. PO_4^{3-} and NO_3^- constitute approximately 7.4 percent of the total (i.e. soluble and insoluble) solids mass. The fraction of the total anions that nitrate and phosphate represent cannot be determined adequately because the analytical method measured only soluble anions and it is known that there are insoluble oxides and hydroxides that are not assayed at this time. The TOC was measured and found to be less than 1.3 weight percent (dry basis) in each core and for the tank as a whole. However, the TOC assay method is believed to be biased low for this waste matrix and individual segment results have been observed to be higher than the bulk value for the tank.

The only significant gamma emitter found in the waste was ^{137}Cs , and it was found at very low levels. No meaningful regional concentrations (hot spots) of radioisotopes or fuel were detected along the vertical axis in either core. The ^{137}Cs concentration was relatively constant between individual core composites and their replicates; however, the concentrations between core 31 and core 33 differed by a factor of 2. In addition, the ^{137}Cs concentration decreased by a factor of nearly 3 as a function of depth between segment 1 and segment 9 of core 33. The major source of radiological activity was ^{90}Sr , which was also at a very low level. The bulk waste temperature in the tank, obtained from a thermocouple tree, ranges between 16 to 20 °C (61 to 68 °F). The radiological activity of tank 241-T-111 waste material was quite low, ranging from 0.3 to 10 mR/hr, measured through the drill string. No significant radiological activity was found in the drainable liquid in the tank or in the water digestion of the samples, but radionuclides were liberated readily in the acid digestion sample preparation, as indicated from the homogenization data. This suggests that ^{90}Sr and ^{137}Cs are insoluble.

Cores 31 and 33 appear to have a T-Plant process flush disposed on top of 224 waste, overlying a 2C waste heel. ^{241}Am and ^{137}Cs decrease substantially as a function of depth, thus their profiles nominally agree with fill histories, although individual batches and process upsets can show characteristics contrary to the general trend. These observations are consistent with the historical information regarding the transfer history, and the ICP element distribution through the segments. Discernable exothermic behavior was detected in the upper segments of both cores 31 and 33, even though the magnitude of the exotherms did not agree with the TOC present. This lack of an identifiable fuel source was attributed to the inability of the persulfate oxidation method to adequately quantitate TOC in this waste matrix. Another hypothesis under investigation is the potential contribution of an exothermic inorganic reaction that has not been identified.

Historical data indicated that 2C and 224 wastes were not expected to give any exothermic response at all, and could not create a propagating hazard. This prediction has been upheld

for 2C wastes as no exotherms were observed. In the 224 wastes, even though some small amounts of oxalate were indicated in the flowsheets, no reaction was expected. Calculations of the bulk waste inventory and inventories for several analytes of interest to the various safety issues [organics (as TOC), NO_3 , ^{137}Cs , ^{90}Sr , plutonium, and water] were made. The calculated TOC by weight percent was smaller than the watch list criterion on a bulk basis, but the energetics results do not reconcile well with that interpretation, and indicate that the organic content in the tank may be disproportionately partitioned between the upper 100 cm of waste and the remainder of the tank. However, it is important to note that the organics concentration, even in this hypothesized enriched layer, may be too low to support a self-sustaining reaction in its present state. Reactions were observed only after all water had been removed from the waste matrix, and water makes up over 75 weight percent of the waste, providing an enormous heat sink to be overcome before reactions can be initiated. Both the historical and analytical data from tank 241-T-111 strongly indicate that the waste lacks the fuel concentration needed to sustain any propagating exothermic behavior or a heat source intense enough to trigger a reaction. None of the other calculated bulk inventory values exceeded any level of concern (see Table 8-1).

Experimental and analytical evidence from tank 241-T-111 waste suggests the risk from organic compounds in this particular Hanford-Site high-level waste tank is acceptable and that a propagating exothermic reaction under current and near-term tank operating conditions is not credible.

Table 8-1. Comparison of Tank 241-T-111 Analyte Values to Safety Issue Criteria.

Analyte	Safety issue criteria ¹	Calculated/measured value
ΔH (dry basis)	-75 cal/g	In excess of -215 cal/g
$^{239/240}\text{Pu}$	50 kg	9.2 kg
Temperature	300 °F (149 °C)	16 °C (60.5 °F)
Heat load	11.72 kw	0.08 kw
Organic content (TOC, Dry basis) (10% sodium acetate equivalent)	3.0 wt% TOC	1.3 wt% TOC (This result is likely biased low)

¹(Lindsey 1986, RHO 1988, Boyles 1992, Reep 1992)

8.1 RECOMMENDATIONS

The following recommendations are made based on the data and analyses presented in this report and the goals of the characterization effort.

- Investigate the potential existence of alternate transfer paths from T-Plant.
- Examine more closely the chemical behavior, reactivity, and composition of Turco decontamination agent.
- Continue to characterize tank 241-T-111 sample matrices at PNL.
- Investigate the possible kinship between tank 241-T-111 and other tanks.
- Research and develop improved assay methods for TOC.
- Investigate observed discrepancy for alpha-emitting radionuclides (especially ^{239/240}Pu and U).

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Appendix A: Analytical Data

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Tank 241-T-111 Core 31 and Core 33 Introduction and Narrative

Introduction

The analyses in this data package were performed by the Westinghouse Hanford 222-S Laboratory or the Battelle Pacific Northwest Laboratory (PNL) under the guidance provided in the "Waste Characterization Plan for the Hanford Site Single Shell Tanks" (WHC-EP-0210) and the "Sampling and Analysis of Ten Single Shell Tanks" (WHC-SOW-91-0006). The quality control for single shell tanks is described in appendix D of WHC-EP-0210. Additional support data can be found in appendix I of WHC-EP-0210. Laboratory operations at 222-S are performed according to the "Quality Assurance Project Plan for the Analysis of Highly Radioactive Samples in Support of Environmental Activities on the Hanford Site" (WHC-SD-CP-QAPP-002) unless superseded by the waste characterization plan, appendix D, the associated SOW, or the Technical Project Plan (TPP).

Tank 241-T-111 (referred to as T-111 in the remainder of this package) is a single shell tank built in 1944 with an operating capacity of 500,000 gallons. T-111 received 488,000 gallons of "2C" and "224" type wastes through February 1976, and is classified as EHW (extremely hazardous waste). During early remediation efforts, ending in 1978, nearly 25,100 gallons of liquid was pumped out of T-111. The "2C" waste was from the second decontamination cycle of the bismuth phosphate process at the B and T plants. This waste consists of the liquid remaining after precipitation of the plutonium. The "224" waste was from the final decontamination and concentration stage of the bismuth phosphate process. In this stage, first the by-products and then the plutonium are precipitated with lanthanum fluoride. Historical data indicates the major components and estimated concentrations of these waste streams are as follows:

Component	224 (mol/L)	2C (mol/L)
Bi	0.0062	0.0092
Cr	0.0009	0.0025
F	0.31	0.22
Fe	--	0.023
oxalate	0.028	--
K	0.26	--
La	0.0014	--
Mn	0.0046	--
Na	1.75	2.04
nitrate	1.06	1.27
hydroxide	0.59	--
phosphate	0.049	--
Si	--	0.37
sulfate	--	0.62

Based on prior knowledge, cores from T-111 were expected to contain 8.5 segments. The first segment is normally the partial segment. Three cores were obtained from T-111 - cores 31, 32, and 33. Core 32 was not analyzed as the recovered material, all liquid, was considered not representative of the tank. No drainable liquids were recovered from cores 31 and 33. Drainable liquids are defined as quantities greater than 25 mL. Less than 25 mL quantities are blended back into the sample. Homogenization tests were performed on segments 1, 3, 5, 7 and 9 from core 33. Two composite samples were prepared for core 31 and core 33. Sample identification is obtained from the "sample point" section on the analytical card. A sample from core 31, segment 1 is designated "T111-C31-S1". Similarly, a sample from composite 1, core 33 is designated "T111-C33-C1", and a homogenization sample from core 33, segment 5 is designated "T111-C33-S5H".

All of the segments, with the exception of segment 9 of each core, were a homogeneous brown-black mud-like material. The bottom half of segment 9 had a white layer. The homogenization test for segment 9 used the entire segment (both phases) from core 33. Additional analysis (acid digest, water digest, ICP, GEA, TA and IC) were requested for the white layer on material from core 31. This sample is designated "T111-C31-S9B". All samples from T-111 exceeded the six month holding time limit.

Fifteen samples were sent to PNL. Volatile Organic Analysis (VOA) were requested on odd numbered segment samples for both cores and rheology on even numbered segment samples for core 31. The VOA requested for segment 9 is on the white phase only. Samples from both composites from each core were submitted for semi-VOA, Extractable Organic Halides (EOX), As, Se, Ni-63, Pu and U isotopic analyses. These samples and the requested analyses are as follows.

VOA:	core 31, segments 1, 3, 5, 7, 9.
	core 33, segments 1, 3, 5, 7, 9.
Semi-VOA, EOX, As, Se:	core 31, composites 1, 2.
	core 33, composites 1, 2.
Ni-63, Pu and U isotopic:	core 31, composites 1, 2.
	core 33, composites 1, 2.
VOA, semi-VOA:	hot cell blank.
	field blank.
Rheology & Physical:	core 31, segments 2, 4, 8.

The required QC is listed in table D-6 of appendix D, WHC-EP-0210. In summary, the requirements are:

- one laboratory control standard per analytical batch,
- one blank per analytical batch,
- one matrix spike per core or per matrix change,
- 100 % duplicates on all homogenization test samples and core composite samples,
- one duplicate per analytical batch for direct segment samples,

-- a duplicate to verify each detected exotherm for DSC analysis.

The exceptions to the above guidelines are:

- % water is always ran in duplicate,
- Sr-90, Se-79, Tc-99, I-129, Pu and Am have a spike or carrier added to each sample, so no additional matrix spikes are required,
- GEA and pH do not require a spike,
- a matrix spike for Np is requested on each sample,
- ICP, HYAA, CVAA, and IC require additional, method specific QC. Instrument calibration and check standards are run according to specific procedure protocols.

Sample analysis are repeated at least once if the spike recovery is outside of $100\% \pm 25\%$ (provided the spike concentration is at least 25 % of the sample concentration), or the duplicates have a relative % difference greater than 20 % (provided the analytical results are greater than 5 times the detection limit). If the original problem remains after one re-run, and the chemist has no explanation or "fix", additional re-runs are not required. These situations should be described in the "Analytical Batch Summary Sheet" described below. The entire batch does not have to be re-run unless the standard or blank for the batch failed.

A "Characterization Change Notice" was implemented during the T-111 analysis. The intent of the form is to document minor changes in analyses and deviations from the SOW or waste characterization plan. These are attached at the end of the summary section.

A "Chemist's Batch Summary Sheet" form was introduced for T-111 sample analysis and appears at the end of the analytical batch. Since this form was introduced after analysis started, one may not accompany each sample batch. The intent of this form is to document the chemist's initial data evaluation, and to provide a means of obtaining descriptive results from the chemist and chemical technologist on each sample batch. While not required as part of the final package, these forms will aid in preparing this narrative and provide insight into some of the problems encountered during sample analysis.

All analytical data for the package appears in a summary section. The summaries are lotus spread sheets intended to aid in review of analytical data, and may not include all of the associated laboratory control standards, dates, or laboratory identification numbers (J numbers). The spread sheet summaries display analytical results, blank data, detection limits, LMCS recoveries and spike recoveries, and using this data, calculates averages for duplicates, the relative percent difference between the duplicates, a mass and charge balance (described below), a ratio of the result to the detection limit, and various inter-comparisons throughout the summary. Secondary spread sheets are supplied that compare the core composite and homogenization duplicate and average results. Since a spike is not required for each sample, spike data is copied with each associated sample. "Less than" values are not used in any of the spread sheet calculations.

All analytical results are "wet weight" values, based on the actual extrusion weights. To convert to dry weight, divide the "weight wet value" by the weight fraction of solids ($100 - \% \text{water} / 100$). All the preparation blanks and detection limits are reported in the same units as the sample results by assuming a typical sample size. This is done to provide a direct comparison of the background and degree of contamination present in the method to the sample results. Since the sample size is estimated, a "less than" value provided for a sample could be slightly lower than the given detection limit or blank value.

A charge balance is calculated for each core composite sample in the analytical summary. The charge balance is calculated by ratioing the equivalent charge of the cations to the anions. As an example, the equivalent charge of sulfate is calculated as follows:

$$\frac{\text{average ug/g sulfate}}{\text{milliequivalent weight of sulfate}} \times 1000$$

Only the major components are used for these calculations, those with a concentration greater than 1000 ug/g. The cations used are Ca, Cr, Fe, Mn, K, Na, Bi, and La from the acid digested ICP results. The anions used are fluoride, nitrate, phosphate (calculated from the P on the acid digested ICP result), sulfate (calculated from the S on the acid digested ICP result), and acetate (calculated from the TOC results). The ratio ranges from 1.3 - 1.5. Proper incorporation of hydroxide would probably improve these ratios, since there is a relatively large uncertainty in the assumed molecular speciation model.

A mass balance is calculated for each core composite sample in the analytical summary, using an "oxide model" as the basis for the calculations for metals and the anions were present in their normal forms. The mass balance is the sum of the weight fractions of the major constituents (those with a concentration greater than 1000 ug/g). As an example, the weight fraction calculation for CaO is as follows:

$$\frac{(\text{ug/g of Ca} \times \text{molecular weight of CaO})}{(\text{atomic wt of Ca} \times 1000000)}$$

The components used in calculating the mass balance are Bi, Ca, Cr, Fe, Mn, Na, U, F, nitrate, phosphate, sulfate, water, K, and La. It was assumed that all but K, Na, and F were present as oxides. The ratio ranged between 0.93 and 0.96.

SECTION I: EXTRUSION AND SAMPLE DATA

Visual

A summary of the extrusion data, entitled "Physical Properties Summary", is provided with the segment data. This is a compilation of all the recoveries for each segment of each core, with an associated percent recovery, penetrometer reading, and density. Photographs of the extruded segment are included in the data package. All volumes given that are less than 180 mL (100 % recovery) are estimates provided by the hot cell chemist. Segments marked with an asterisk on the summary sheet were recovered from sample

containers with the valves left in the "open" position (the waste was thick enough to remain in the container, even though the valve was not closed). The extrusion data for core 32 will be included in the core 31 package.

Core 31

- Segment 1: The sampler was almost empty. It contained about 50 mL of homogeneous, black-brown, low viscous solids. The entire sample was used in VOA and DSC/TGA sampling. There was a tiny amount of liquid, not enough to observe characteristics.
- Segment 2: The sampler was completely full of homogeneous, dark brown to black solids with the consistency of swamp mud. There were no drainable liquids.
- Segment 3: The sampler was completely full of homogeneous, dark brown solids that had the consistency of swamp mud. There were no drainable liquids.
- Segment 4: Most of the sampler was full of the same dark brown homogeneous solids. The top 1/8 of the extruder was filled with a more "liquidy" solids than the rest. There were no drainable liquids.
- Segment 5: The sampler was completely full of the same dark brown homogeneous solids and contained no drainable liquids.
- Segment 6: This sampler was empty, the valve was left in the open position.
- Segment 7: The sampler was completely full of the same dark brown homogeneous solids and contained no drainable liquids.
- Segment 8: The sampler was completely full of the same dark brown homogeneous solids and contained no drainable liquids.
- Segment 9: The sampler was completely full of solids, the same dark brown solid material at the top, but gradually whitened down the segment. While the color varied, the consistency of the material was the same as all the other segments in this core.

Core 32

- Segment 1: Contained about 50 mL (58.70 g) of "dark dishwater grey aqueous" liquid with about a 2 mm layer on the surface. There was a layer of bubbles covering the thin top layer, and adhering to the sides of the jar. No solids were collected.
- Segment 2: Contained about 170 mL (88.35 g) of dark grey liquid with dark grey particles suspended in the liquid. There were only minimal solids (enough for a VOA sample) that had the consistency of loose mud or pudding and was dark brown/black in color.
- Segment 3: Contained 180.90 grams of dark grey aqueous material. No solids were present. No volume was recorded.

- Segment 4: Contained 182.48 grams of dark grey aqueous material. No solids were present. No volume was recorded.
- Segment 5: Contained 191.05 grams of dark grey aqueous material. No solids were present. No volume was recorded.
- Segment 6: Contained 185.52 grams of dark grey aqueous material. No solids were present. No volume was recorded.
- Segment 7: The sampler was empty, the valve was left in the open position.
- Segment 8: The sampler was empty, the valve was left in the open position.
- Segment 9: Contained 158.24 grams of liquid, with about 150 mL of aqueous phase and 8 mL of NPH.

No samples were taken from core 32 as the material was judged to be non-representative of the material in the tank when compared to cores 31 and 33.

No photographs were taken for segments 1, 3, 4, 5, 6, 7, 8, 9.

Core 33

- Segment 1: The sampler was completely full of nearly black homogeneous solids, and contained no drainable liquids.
- Segment 2: The sampler was completely full of the same nearly black homogeneous solids, and contained no drainable liquids.
- Segment 3: The sampler was completely full of the same nearly black homogeneous solids, and contained no drainable liquids.
- Segment 4: The valve was left open on this core assembly. The extruder was about 75% full dark brown/nearly black homogeneous solids, and contained no drainable liquids. Color was slightly lighter than previous segment.
- Segment 5: The valve was left open on this core assembly. The extruder was about 7/8 full of the same dark brown homogeneous solids, and contained no drainable liquids.
- Segment 6: The sampler was nearly full of the same dark brown homogeneous solids, and contained no drainable liquids.
- Segment 7: The sampler was nearly full of the same dark brown homogeneous solids, and contained no drainable liquids.
- Segment 8: The sampler was nearly full of the same dark brown homogeneous solids, and contained no drainable liquids.
- Segment 9: The sampler was completely full of solids, the same dark brown solid material at the top, but the bottom 5-6 inches was light colored. While the color varied, the consistency of the material was the same as all the other segments in this core. The segment

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was divided into two parts, one was the bottom 5 inches of the segment which contained most of the white material. A VOA sample was taken from the top, and a DSC/TGA taken from the bottom. These samples were later combined for the homogenization test.

A field blank and a hot cell blank were also taken for T-111.

Sub-samples for T-111

The following numbers are the "J" numbers assigned as tracking numbers by the 222-S laboratory. The analytical batches may contain information from other sample numbers because of the way the samples were grouped for analysis. Results from other samples should be ignored, since they will be included in the data package for that specific segment.

	CORE 31	CORE 33	CORE 33 (Homog. Tests)
Segment 1	J308, J309	J319, J320	J396, J397, J398, J399
Segment 2	J310	J321, J323	NA
Segment 3	J311	J324	J400, J401, J402, J403
Segment 4	J312	J325	NA
Segment 5	J313, J315	J326, J327	J404, J405
Segment 6	none (empty)	J328, J329	NA
Segment 7	J315, J316	J330	J406, J407, J408, J409
Segment 8	J317	J331	NA
Segment 9	J318	J332, J333	J410, J411
Segment 9B	J412, J413, J414	NA	NA
Core 31 composite 1	J415, J416, J417, J424, J425, J426, J432, J433, J438, J439, J440, J447, J448, J449, J455, J456, J457, J464, J465, J466, J472, J478, J479, J480, J493, J506, J507, J608, J609, J610, J611, J630, J631, J632, J641, J642, J643		
Core 31 composite 2	J418, J419, J427, J434, J441, J450, J459, J467, J475, J485		
Core 33 composite 1	J420, J421, J428, J429, J430, J435, J436, J442, J443, J445, J451, J452, J453, J460, J461, J462, J468, J469, J470, J476, J487, J488, J489		
Core 33 composite 2	J422, J423, J431, J437, J446, J454, J463, J471, J477		

Hot cell blank

J494, J495, J496, J498, J499, J500, J502, J503, J504,
J506, J508, J513, J517

SECTION II: SEGMENT ANALYSIS

TGA

Thermogravimetric Analyses and Differential Scanning Calorimetry (DSC) are performed on every visible phase of the waste, prior to homogenization, to evaluate the thermal properties of the waste. The TGA results give a measure of the weight loss with increasing temperature. The result obtained from TGA is a weight % water. The TGA % water (percent weight loss at 100 degrees C) may vary considerably, due to a combination of the small sample size and sample heterogeneity. The procedure number for DSC, T042 A-01712F, has been changed to LA-514-113, Rev. A-0. The procedure number for TGA, T045 A-00712F, has been changed to LA-560-112, Rev. A-0. These procedures are routine Analytical Operations now, rather than Process Chemistry procedures.

The TGA results were 5-7% higher than the gravimetric wt % water, and ranged from 70-87%. Where duplicates were ran, there was good agreement, and results were consistent over all segment samples

% Water

More representative % water measurements are obtained with the gravimetric measurements, also reported in the summaries. Duplicate % water analysis were requested for all segment samples. However, the duplicates for segments 2, 3, 4, 5, 8, 9 from core 31 and segments 1, 3, 4, 5, 7, and 9 from core 33 were not requested until one month after the original samples were run. These latter values are added to the summary sheet, but due to the summary sheet format, the LMCS standard results are not included. The duplicate values for segments 2 and 4 from core 31 (32.60% and 59.60%) are probably low due to the sample drying out between analysis, or from a non-homogeneous "soupy sample" that would cause uneven sampling. There is no more sample for a re-run. However, the original sample results and the TGA agree quite well. There was no sample for a duplicate analysis of segment 8 from core 33.

DSC

DSC is used to identify the potential of exothermic reactions from the waste upon heating. In all cases, the identified exotherms were 3 to 4 times smaller than the detected endotherms. The calibration frequency of the instrument is determined by the chemist and is based on the instrument performance on laboratory standards.

Core 31: Exotherms were detected on the sample and duplicate for segments 1, 2, and 3. A very small exotherm, nearly an order of magnitude smaller, was detected for segment 4, and was not verified with a duplicate. No exotherms were detected for segments 5, 7, 8, 9, and 9B (the white layer). The temperature ranges for the exotherms were between 200 and 405 °C, with the energy around 300 J/g. The small exotherm had an energy of 55.7 J/g.

Core 33: Exotherms were detected on the sample and duplicate for segments 1 and 2, and only the sample for segment 3. The duplicate did not show an exotherm. The temperature ranges for the exotherms were from 179 to 438 °C,

with the energy around 300 J/g. The exotherm for segment 3 was small, about 50 J/g. No exotherms were detected for segments 4-9.

Particle Size

Particle size analyses are performed on every segment from every core to support the evaluation of potential waste retrieval methods. Small aliquots of the wastes were suspended in a water matrix using sonification before measuring the particle size by laser technology. The data are summarized in a table and four plots of probability number and probability volume density and distribution graphs. The procedure number for particle size analysis listed in the Technical Project Plan (WHC-SD-CP-TP-070, rev 0) is incorrect, and should be T044-A-01712F.

Penetrometer

Penetrometer readings are taken for each segment at extrusion. All of the segments were soft, with readings <100 psi.

Segment 9B (white layer)

In addition to the above analyses requested for each segment, ICP, IC, GEA, and TA were requested on the white portion of segment 9 of core 31. The sample was taken after the white layer had been homogenized. The ICP and TA resemble the homogenized segment data, and the IC resembles the core composite data. The spike recoveries for the ICP were biased low. There were no exotherms detected, and the % water was 70 %, about 5-10 % lower than the other segments.

General Comments

Because of the qualitative nature of TGA, DSC, particle size, and penetrometer analyses, no detection limits or precision and accuracy statements are required.

SECTION III: HOMOGENIZATION TEST ANALYSES

Introduction

Waste tank samples have a wide range of physical characteristics. Methods for homogenizing waste samples are limited by their applicability to hot cell operations and how well they lend themselves to remote operation and decontamination for reuse. Homogenization tests have been incorporated into the single shell tank characterization program to obtain estimates of the errors resulting from homogenization techniques employed during analysis. The evaluation of this data will support decisions made using the data and identify areas where homogenization procedures may be improved.

Homogenization Tests

Homogenization tests are performed on segments. After the segment has gone through the homogenization procedure, two sub-samples are taken from two different (top/bottom, left/right) locations in the homogenized segment. Each of the sub-samples are analyzed in duplicate to obtain estimates of the

analytical error. The differences in the results obtained on the two sub-samples will provide homogenization error. A statistical analysis (beyond the scope of this narrative) of the variance must be performed to differentiate between the analytical and homogenization errors. Each homogenization sample was analyzed by GEA, ICP, and TA on an acid digested sample. The acid digestion is the same as that used for normal ICP analysis.

Sub-samples

Homogenization tests were performed on segments 1, 3, 5, 7, and 9 from core 33. See the listing above for the J numbers assigned to the homogenization samples. The results are compiled in the summaries entitled "T-111 Homogenization Analyses" and "Homogenization Test Summary". One spike was considered sufficient for the homogenized sample data. A comparison of the RPD's of the duplicate results of each sample to the RPD of the averages of the two samples (see summary) shows virtually no difference for those elements well above their detection limits. This tank probably is not a good test of homogenization procedures, as the segment material was very uniform at extrusion. Even the white material in segment 9 was chemically similar to the rest of the segments, with the exception of segment 1. Segment 1 has much higher concentrations of Mn, Pb, Ag, and Zn when compared to the other segments, while Bi and La are much lower. The spike for the gross alpha on segment 5 was consistently high even after several re-runs.

SECTION IV: CORE COMPOSITE ANALYSES

Sample Preparation

Samples are prepared for core composite analyses using an acid and water digestion, and a KOH fusion. ICP and HYAA are run on the acid preparation, ICP, IC, nitrite by spectrophotometry, pH, ammonia, TOC, TIC, carbon-14, and tritium on the water digestion, and ICP and the other radiochemical analyses on the fusion preparation. The KOH fusion is contaminated with Ni from the crucibles used. LMCS standards for the acid digestion go through the sample preparation procedure. Standards for the water digestion and fusion are not run through the sample preparation procedures.

ICP

The ICP data is reported using a CLP software package from WARD Scientific Ltd (WARDS), and is used to generate the spread sheet summaries instead of the analytical cards. The calculations on the analytical cards are preliminary calculations performed by the instrument technician to access instrument operation and are not used in the final report.

WARDS utilizes the concept of a sample delivery group (SDG), and batches spike, duplicate, instrument control standards, blanks, serial dilutions, interference checks, and narrative information for each sample group. If the result is below the detection limit, the CLP report format will return a 200% RPD for duplicates and a 100% for serial dilutions. On all pages except the "duplicate" page (form VI-IN), any values less than the detection limit are replaced with the instrument detection limit, and should be interpreted as a "less than" value. The duplicate page (form VI-IN) reports what the instrument measures, and may include negative numbers. Since it is this page

that is used to generate the "result 1" and "result 2" columns in the summary sheets, negative values are included.

On the page in the WARDS package designated "COVER PAGE - IN", the answer to the third question, "...were raw data generated before application of background corrections?" has been changed to "no" based on the chemist's interpretation. This change does not affect the data or evaluation of the data in any of the WARDS packages generated in support of the SST program. The WARDS package Sample Delivery Group (SDG) number has a "/" suffix, where the * is "N" for no sample preparation, "W" for water digestion, "F" for fusion, and "A" for acid digestion. This was not implemented until after analysis began on T-111, and so most of the WARDS packages will not have this identifier. The case narrative provided with each batch flags areas that would not meet CLP criteria. The WARDS package is configured to report all elements requested in the SOW except Tl, Th, and U, and report the additional elements B, Ce, La, P, Sr, S, and Ti. Form I-IN will not show negative results for individual determinations, while Form VI-IN does. The WARDS results used in compiling the master summaries are taken from the VI-IN form.

ICP analyses are ran on acid and water digests, and a KOH fusion. In the data summaries for the T-111 package, the preparation blank and the detection limit values for ICP have been converted from ug/L to ug/g by estimating the typical sample size and dilution involved in the preparation procedure. The acid and water blank and detection limit results (ug/L) were divided by a factor of 10 because the acid and water preparation procedures dissolve approximately 1 g of original sample in 100 mL of liquid. The fusion blank and detection limit results were divided by 2, as this preparation procedure dissolves approximately 0.5 g of sample in 250 mL of liquid.

The units for the "SA" column (spike added) on form V-IN are "mg/kg" for the pre-digestion spiked acid sample preparation, and "ug/L" for the post digestion spiked water and fusion sample preparations. Spike concentrations must be at least 25% of the sample concentration for proper evaluation.

Failure criteria for an entire WARDS package are under development. Since each ICP run generates results for 32 elements, there will always be some failures on each run. These failures are identified in the narrative associated with each WARDS package. Failure criteria should be based on the absolute failure rate of either spikes, duplicates, LMCS standards, and calibration standards, but tempered by the relative importance of the individual element that failed. For example, LMCS and spike recoveries may be generally high for Na, Ca, and Fe from contamination of the blank and sample during preparation, and Si and B from the use of glass containers during sample preparation. Spike or LMCS failures noted for these elements should not invalidate the entire WARDS package.

Several factors noted in the WARDS packages for T-111 create low spike recoveries and were noted by the ICP chemist. Silver recoveries are commonly low due to the precipitation of Silver Chloride. Poor recoveries of Iron, Magnesium, and Calcium accompany high Preparation Blank values, but the correlation is poor. Spike failures for major elements are frequently caused by a high element concentration in the sample. When the added spike concentration is insignificant compared to the concentration of the element present in the sample, a failure generally occurs.

The major elements detected by ICP analysis of the acid digestion were Ca, Cr, Fe, Mn, K, Na, Bi, La, P, and S (all greater than 1000 ug/g). Cr and Pb concentrations ($1.86E+3$ and $4.75E+2$ ug/g respectively) would both exceed the 100 ug/g TCLP limit, assuming 100% leachability. The WARDS package performs inter-element corrections for Al, Ca, Fe, Mg, and Cr. Calcium and magnesium contamination often occurs as a result of the powder on the gloves used by laboratory personnel. The ICP analysis of the fused sample is used to evaluate the completeness of the acid digestion procedure, and to identify any acid insoluble compounds. The K results in the WARDS package often over ranges on the fusion, giving the impression that no K was in the sample. Ratios of the ICP results for major elements appear in the data summaries, and are used to estimate consistency and speciation. The fusion/acid ratio shows good agreement for Al, Fe, Na, P and Sr. Si and Ti are an order of magnitude higher in the fusion, while Bi is slightly higher in the acid digestions. Based on the ratio of the acid and water ICP data, about 10 % of the Cr is water soluble (CrVI). Most of the Na is soluble, but only 50 % of the phosphate, and essentially none of the Mn is soluble. There was no La reported for the water digested composite LMCS standard for core 33, and no Zr detection limits reported for the core composite samples. The Si results for ICP on all T-111 analyses are known to be biased low due to an undetermined interference.

CVAA and HYAA

Cold vapor atomic absorption (CVAA) was used for Hg analyses. The Hg results for T-111 ranged from 1.02-1.88 ug/g. The spike for core 31 was high (122%) and low for core 33 (82%). However, both were within the plus or minus 25% limits. All RPD's were less than 13%. Due to instrument and sample matrix problems, the As and Se elemental analyses were performed at PNL by graphite furnace AA.

Anion and Wet Chemical Analysis

Anions were determined on a water digestion of the sample. Ion chromatography (IC) was used to determine F, Cl, nitrate, nitrite, phosphate, and sulfate. All duplicates were within limits (<20 % RPD) and the results were consistent over all the composite samples. Post digestion spikes are used because of the high concentrations of the anions. Nitrate, phosphate, and F concentrations were high, consistent with the historical data available on T-111 waste. Spike recoveries were biased low for 3 of the 4 composite samples, but within the 75-125% limits. The S determined on the acid ICP, converted to sulfate, agrees very well with the sulfate determined by IC, while the same comparison of P with the phosphate shows the converted P a factor of 2 higher than the phosphate. This indicates that only 50 % of the phosphate is soluble. The pH is very consistent at about 10, while only "less than" values were obtained for ammonia. All the TIC results were near the detection limit, with good reproducibility and spike recoveries. TOC results were nearly an order of magnitude higher (2000-3000 ug/g). All analytical units were changed to ug/g for the data summaries, and may not agree with what is on the analytical card. All cyanide results obtained were "less than" values. Both the % water and TGA results were between 75 and 80 % with good precision over all the core composite samples.

Radiochemical Analysis

Most of the radionuclides are determined on the fusion due to the acid insolubility of actinide and Sr compounds. Tritium and C-14 are determined on the water digestion, and Ni-63 (analyzed at PNL laboratories) is determined on the acid digestion. There are indications that I-129 is lost during the acidification of the fused sample, which will require the development of an alternate digestion procedure for future work. In the cases where two spike recoveries are reported in the analytical summaries, one under the other, the spike recovery is used to correct for chemical yield. The second spike (bottom) corresponds to the duplicate result. Pu-238 is reported as part of the Pu-239 analysis, using the spike data from the Pu-239 procedure. Cm-244 would be reported only if seen on the Am-241 mount. The relative counting error reported is based on gross counts, and will under-estimate the counting error in those samples close to the detection limit (where the gross counts and the background counts are nearly the same). Calibration data for GEA labeled as detector "14" is data for detector "4".

During calibration updates, the new calibration file name for individual detectors have a "1" added (4 to 14) to avoid erasing the old calibration data prior to validation of the new calibration data. For T-111, the new calibration file name was not changed back to "4". Calibration of the alpha proportional counters (APC) is performed as follows. A standard source is prepared by the standards laboratory and used to calibrate one of the three detectors. Since the calibration standard is not durable enough for daily use, a secondary source is prepared and the d/m value of the secondary source is assigned using the calibrated detector. The secondary source is counted daily on all three APC's and tracked using the LMCS system. The detectors pass the daily check if the calculated efficiency for each detector is within $50\% \pm 0.25\%$ (100% "recovery"). The LMCS data for the APC's and EDP codes are listed in the "Precision and Accuracy Statement" section.

Poor spike recoveries were obtained for uranium on core 33, but the data across all the core composite samples was consistent (between 2000-3000 ug/g).

Total alpha and total beta RPD's were normally under 5% with good recovery on the spikes. The total alpha results were twice the sum of the Pu and Am results, indicating 1) the potential presence of undetermined alpha emitters, 2) beta cross-talk (unlikely due to the beta activity present), or 3) incorrect Pu or Am recoveries (incomplete exchange with the spike). This last scenario is unlikely due to the consistency of the ratios, and the excellent agreement between the Am-241 results obtained by gamma spectroscopy and alpha spectroscopy. The ratio of the total beta to the sum of the Sr/Y and Cs-137 results show good agreement (.991 to 1.00) for the activity balance of the beta emitters. When sample "J-470" was rerun for poor spike recovery, a blank was not included in the analytical batch.

Sr-90 spike recoveries were consistently 90-95%. Results for core 31 were around 7 uCi/g while core 33 was around 3.5 uCi/g. These results vary more than the elemental Sr results from the fusion ICP, where the results are consistent across both cores (280-310 ug/g). Tc-99 results were low, within a factor of 5 of the detection limits but spike recoveries were adequate (67-74%). The results for Se-79, H-3, and C-14 were at or below detection limits, while spike recoveries ranged from 85-95%. No standard material is available

for Se-79. The tritium procedure LA-218-113 was changed to LA-218-114 for T-111 analysis. This procedure uses microdistillation columns in place of the normal glass distillation equipment.

The Pu spike recoveries ranged from 50% to 90%, while Am recoveries were poor, 10-20%. No Pu-238, Np-237 or Cm-244 were present above detection levels. Spike recoveries for Np-237 were consistently low at around 70%.

Am-241 and Cs-137 were the only isotopes that were consistently found at quantifiable concentrations by gamma spectroscopy. No spikes were requested for gamma spectroscopy. As stated above, the beta activity based on the Cs-137 results from gamma spectroscopy agreed with the gross beta determination, and the Am-241 results from gamma spectroscopy agreed with the alpha spectroscopy results for Am. I-129 results were all "less than" values, therefore no counting error could be determined. The preparation blank for I-129 for core 33 was invalid based on spike recovery, but was not re-run as all results were "less than" values. The spike recoveries ranged from 39% to 57%.

Physical Measurements

DSC, TGA, and weight % water was performed on each core composite. Exotherms were observed (in duplicate) for both composite samples from core 31, but no exotherms were observed for core 33. This is not surprising, as the exotherms were small on the segment analysis and were done prior to homogenization. TGA and % water results are in the same range as found on the segment samples, 75-85%, with the TGA results being consistently higher than the % water.

TCLP Results

A TCLP extraction was performed on the waste using a TCLP procedure scaled down a factor of 10 for radioactive samples. The matrix spike was added before the TCLP extract was preserved and acidified (per SW-846 procedure), recognizing that possible precipitation of the spike material could result in low spike recoveries. In order to meet the duplicate, spike, and blank requirements of appendix D, the following sampling and digestion scheme was used on T-111. Composite 1 and composite 2 from core 31, composite 1 from core 33, and a blank were run through the TCLP digestion procedure. The composite 1 TCLP digests from core 31 and core 33 were split into two sub-samples, and one sub-sample from each digestion was spiked with the TCLP metals. All sub-samples were acidified. Each sub-sample was acid digested in preparation for analysis, with the unspiked sub-samples from both cores again prepared in duplicate (split into two samples prior to acid digestion) to determine analytical error. The composite 2 and blank TCLP digests from above were acidified, acid digested and submitted for analysis (no spike or duplicate measurements).

The RPD's were high for As, Ba, and Pb, but these analytes were present in concentrations less than 6 times the detection limit. Cd was the only element with spike recoveries within acceptable limits (greater than 75 %). A comparison of spiking procedures is being performed on the next single shell tank (C-110). Cr and Se both exceeded the TCLP limit. Good agreement was obtained for those elements well above the detection limits.

Field Blank and Hot Cell Blank

Field blanks are prepared by filling a sample container in the field, shipping it to the labs with the samples, extruding in the hot cell and collecting as drainable liquids. The hot cell blank is a rinse of the extruder tray. The required analysis for these samples are ICP, GEA, HYAA (As, Se), CVAA (Hg), TA, TB, TOC, IC, and VOA and semi-VOA. The raw data for the field blank and the hot cell blank is included at the end of the core 31 data package. No duplicate samples or spikes were required on the blanks. The ICP was ran on direct sample, so no corrections were made on the preparation blanks per detection limits. The ICP LMCS standard did not include Sr. There was insufficient sample for duplicate ICP analysis for the hot cell blank.

Hot cell blank: The major constituents in the ICP analysis were Na at 5 ug/mL, B at 1.5 ug/mL and Si at 4.75 ug/mL. All the rest were less than 1 ug/mL. The balance of the analytes determined (F, Cl, NO₂, NO₃, SO₄, PO₄, As, Se, Hg, TOC, TA, TB, and GEA) were "less than" values.

Field blank: The major constituents in the ICP analysis for the field blank were similar to the hot cell blank. The Na was at 8.07 ug/mL, B at 1.76 ug/mL, and Si at 4.47 ug/mL. The rest of the constituents were less than 1 ug/mL. Results greater than the detection limits were obtained for F, NO₃, and PO₄ on the IC, TB, and Cs-137 on the GEA. These levels of contamination are insignificant when compared to the concentrations of these analytes in the T-111 samples.

Accuracy and Precision Statements

Accuracy and precision estimates for the procedures used in analyzing T-111 core samples were compiled from the 222-S Laboratory Measurement Control System (LMCS) data base over the period of April 1, 1992 through July 22, 1992 (the time period that the samples were ran in the laboratory). The average percent recovery and standard deviation for each analyte are determined from multiple analysis of standards containing those constituents. Each analyte and analytical system has a specific EDP code. A different EDP code is used for each parameter, instrument, method, or standard used in the system. When more than one EDP code is provided, then more than one analytical system was monitored. If no data was available for an EDP code for the time frame of interest, the historical values based on earlier data has been provided. In these cases, the number of measurements noted as "n" is recorded as "0". Historical data typically is based on 50 measurements.

No standard Se-79 material is available from commercial suppliers of isotopes for the preparation of a standard. The LMCS standard data for DSC only indicates that an exotherm is detected, and 100 % recovery is noted. The results do not reflect errors caused by sample matrices, or special sample preparation procedures used prior to applying some of the methods.

The accuracy and precision estimates for T-111 are as follows.

<u>Method</u>	<u>Analyte</u>	<u>EDP</u>	<u>n</u>	<u>Average % Recovery</u>	<u>Relative Std. Dev.</u>	<u>Comments</u>
ICP						
LA-505-151	Al	S102	46	96.7	4.9	Historical
	Sb	S104	50	100.2	3.0	
	As	S106	5	99.6	2.5	
	Ba	S108	35	96.9	2.6	
	Be	S110	50	102.3	3.2	
	Cd	S116	21	98.5	3.5	
	Ca	S118	42	96.5	3.5	
	Cr	S122	56	100.3	3.0	
	Co	S124	6	98.6	2.0	
	Cu	S126	18	96.5	1.8	
	Fe	S130	39	98.5	2.7	
	Pb	S134	35	98.3	4.7	
	Hg	S138	30	98.1	3.7	
	Mn	S140	33	95.0	2.8	
	Ni	S148	31	98.9	2.7	
	K	S152	37	99.3	2.9	
	Se	S156	5	100.1	4.9	
	Ag	S160	17	99.1	3.5	
	Na	S162	49	96.1	2.3	
	V	S182	12	98.9	1.4	
	Zn	S184	36	99.0	2.4	
	Bi	S112	23	100.1	4.7	
	B	S114	9	95.8	3.0	
	La	S132	14	103.2	3.0	
	P	S150	31	96.9	4.2	
	Si	S158	23	94.4	5.1	
	Sr	S164	24	96.3	2.2	
	S	S166	32	100.1	3.2	
	Sn	S174	9	97.1	4.6	
	Ti	S176	13	98.1	2.2	
	Zr	S186	9	101.4	3.8	
	Ce	S120	4	103.9	1.3	

<u>Method</u>	<u>Analyte</u>	<u>EDP</u>	<u>n</u>	<u>Average % Recovery</u>	<u>Relative Std. Dev.</u>	<u>Comments</u>
CVAA						
LA-325-102	Hg	R716	18	102.8	6.0	
IC						
LA-533-105	F	R974	18	101.4	5.9	
	Cl	R972	20	99.0	4.4	
	NO3	R978	22	96.8	3.9	
	NO2	R968	22	101.2	4.9	
	PO4	R976	18	97.5	3.7	
	SO4	R970	21	96.9	3.9	
SPEC						
LA-645-001	NO2	S215	88	99.6	4.0	
TOC/TIC						
LA-622-102	TOC	S223	74	103.8	3.6	
LA-344-105	CO3	S346				
Dist/SPEC						
LA-695-101	CN	S242	29	99.4	3.5	
LA-695-102		S244	9	99.5	2.0	
Dist/Titrate						
LA-634-102	NH3	S235	70	99.6	6.4	
		S236	70	99.6	6.4	
Fluorimeter						
LA-925-106	U	S267	111	100.8	8.2	
Alpha						
LA-508-101	Tot Alpha	S510	62	103.8	8.8	
LA-508-104	APC 1	C011	112	99.9	0.11	
	APC 2	C012	112	100.0	0.09	
	APC 3	C013	111	99.5	0.10	
LA-508-114	Det 14	C142	176	99.7	4.8	
	Det 16	C162	169	103.5	5.3	
Beta						
LA-508-101	Tot Beta	S515	59	98.3	4.4	
LA-508-114	Det 14	C143	176	99.0	3.1	
	Det 16	C163	170	99.7	7.4	
Gamma						
LA-548-121	GEA Cs-137	R901	28	101.9	2.4	
	Co-60	R905	28	100.6	2.3	
Alpha Spec						
LA-503-156	Pu-239	R211	20	99.0	5.3	
	Am-241	R201	31	94.2	8.2	

WHC-EP-0806
WHC-SD-WM-OP-024 ADDENDUM-2, REV 0

<u>Method</u>	<u>Analyte</u>	<u>FDP</u>	<u>n</u>	<u>Average % Recovery</u>	<u>Relative Std. Dev.</u>	<u>Comment</u>
Alpha						
LA-933-141	Np-237	S380	20	72.8	13.1	
Beta						
LA-220-101	Sr-90	S376	30	93.7	6.4	
LSC						
LA-438-101	Tc-99	S363	67	108.0	7.8	
LSC						
LA-365-132	Se-79	no standard material available				
Gamma						
LA-378-104	I-129	S928	16	107.8	9.1	
LSC						
LA-348-104	C-14	R909	26	91.6	7.3	
LSC						
LA-218-113	H-3	R907	10	84.7	4.4	
Gravimetric						
LA-564-101	% Water	S360	63	100.3	1.3	
Thermal						
LA-561-112	TGA	S362	6	98.8	1.4	
		R826	10	98.5	1.9	
Thermal						
LA-514-113	DSC	S230	16	100	0	
pH						
LA-212-103	H+	S348	4	100.6	0.15	

Steve McKinney 9/30/92
Steve McKinney
Senior Scientist



Battelle

Pacific Northwest Laboratories

WHC-SD-WM-DP-024
ADDENDUM 5, REV 0-A

Project Number _____

Internal Distribution

Date September 18, 1992

To SG McKinley

From MW Urie *MW Urie*Subject GFAA (As, Se) Analytical Results for T111 (Core 11 & 13)

Graphite Furnace Atomic Absorption analyses were performed September 17 and 18 on the SST samples from Tank T111 Core 11 & 13 core composites following procedures PNL-ALO-214 (As) and PNL-ALO-215 (Se). The digestions were performed according to PNL-ALO-101 in the Shielded Analytical Laboratory and the analytical work was conducted in the Laboratory 303 using a P-E 5100 AA (WB76757). Results and all supporting data are archived in GFAA File: GF091792.

The ICV/CCV analytical control standards for As and Se analyzed between 99% and 108% recovery. The two pre-digestion blank spikes (or "process control standards") recoveries were 118% & 104% for As, and 57% & 58% for Se; the Se results are flagged with an "N" to indicate that the recoveries are less than 75%. The low control recovery on Se is indicative of a loss of Se during preparation; reanalysis was not performed due to lack of sufficient material. The results reported in the Summary Report may be bias low as much as 50% (i.e., if an adjustment is made for the control recovery, the Se results are 3.0 µg/g "U" instead of 1.5 µg/g "U"). The pre-digestion spiked samples for As recovered well (i.e., 95% & 81% at 5x dilutions), whereas the spiked samples for Se showed no recovery (i.e., at 10x dilutions) indicating a severe matrix interference.

The results for the samples, duplicates, spikes and controls is presented in the attached GFAA Summary Report.

Concur: *Josiah B. Brown* 9/23/92

WMC-SD-WM-DP-024
ADDENDUM 5, REV 0-A

GRAPHITE FURNACE AA ANALYSIS SUMMARY REPORT -- BANK 1111 CORE 31/33

Analyte	Sample ID#	PMT ug/g	01				02				01002 RPD	01000 ug/g	03				04				05			
			Sample ug/g	Post Spike Rec	Flags C B	Digst. ug/g	Sample ug/g	Post Spike Rec	Flags C B	Sample ug/g			Post Spike Rec	Flags C B	Sample ug/g	Spike Rec	Spike True ug/L	Control Meas. ug/L	Rec					
As	C31-comp1	92-08277	3.30	102%	U		3.30	100%	U	U	N/A	0.66	104%	U				4.67	95%			2496	3436	138%
Se	C31-comp1	92-08277	1.50	77%	U	M	1.50	82%	U	U	N/A	0.15	93%	U				*	N/A			2496	1432	57%
As	C31-comp2	92-08279	3.30	100%	U		3.30	101%	U	U	N/A													
Se	C31-comp2	92-08279	1.50	76%	U	M	1.50	72%	U	U	N/A													
As	C33-comp1	92-08281	3.30	97%	U		3.30	92%	U	U	N/A	0.66	111%	U				4.15	83%			2496	2601	104%
Se	C33-comp1	92-08281	1.50	80%	U	M	1.50	81%	U	U	N/A	0.15	78%	U				*	N/A			2496	1441	58%
As	C33-comp2	92-08284	3.30	96%	U		3.10	89%	U	U	N/A													
Se	C33-comp2	92-08284	1.50	64%	U	M	1.40	65%	U	U	N/A													

3-5-93

Notes:

AA5100 PE (9/1/92): IDL >> As = 3.30 ug/L, Se = 0.75 ug/L; CRDL >> As = 10 ug/L, Se = 5 ug/L
 Solid B200 dilution: IDL >> As = 0.66 ug/g, Se = 0.15 ug/g; CRDL >> As = 2 ug/g, Se = 1 ug/g
 The analytical spike is 20 ug/L for As and 10 ug/L for Se.
 ICF/CCV used during analysis: As, Se -- ICF ICF2(1200)
 The pre-digestion spike is 25 ug/L for both As and Se.
 Procedures: PM1-A10-214 (As), PM1-A10-215 (Se); NAME: GFAA PE 5100 0076757

Flags:

U = (Analyte) < IDL -- Note the value reported for the sample is the IDL.
 B = IDL < (Analyte) < CRDL
 M = Pre-digestion Spike Recovery not within control limits (75-125%).
 U = Post-digestion Spike recovery not within control limits (50-150%).
 * = RPD not within control limits.

WHC-EP-0806

WHC-SD-WM-DP-024
ADDENDUM 5 REV 0A

Susan McKinley
October 9, 1992
Page 2

-A

The relative percent difference (RPD) is calculated with sample 92-08280-H2 and its duplicate to indicate the overall analytical precision (3% for ^{63}Ni and 2% for ^{65}Ni).

A blank spike and matrix spike were prepared using ^{63}Ni ; no NIST traceable ^{63}Ni was (or is) available for spiking purposes. The blank spike recovery is 95%. The matrix spike recovery is 91%. Thus the average batch bias is -7%.

A:\WP51\T111C313.RPT

-A

RADIOCHEMISTRY STANDARDS AND CONTROLS
Nickel-63 and Nickel-59
Supporting Documentation

This page indicates the location for supporting documentation for radiochemical analyses performed in support of ⁵⁹Ni determinations.

Detector Controls

- LEPS detector background, control counts, and efficiency determination, see control and background files for M&TE # Diode A, Diode B, and Diode D.
- LSC detector background and control counts, see instrument log, Packard 2250XL, Lab 54, Bldg 325.

Solution standards:

- ⁵⁹Ni standard #54124-16-0, LRB 54124, page 16, Bldg. 329, room 10.
- Nickel (stable) carrier solution for yield determination, see LRB 53293, page 30.

Flourescence on the ⁵⁹Ni X-ray peak

- ALO office, 101SY ⁵⁹Ni report

Performance checks of pipets and balances, see LRB 53647 pg. 33, 35, & 36 and LRB 54371, pg. 41.

cr34cl12.sup

TABLE 11a. SST Core 31 & 33, Radiochemistry Data
Pu-238, Pu-239/240, Pu-241, Pu-242

Sample ID	TOTAL ALPHA Pu		Pu-238				Pu-241		Pu-242	
	U/L/g	error	Mass Percent	Pu-239	Pu-240	Pu-241	Pu-242			
Core 31 & 33										
92-08276-N-1 f/amp	4.22E-010	7	0.005	96.6924	3.2230	0.0373	0.0136			
92-08276-N-2 f/amp	0.14E-010	7	0.005	96.7474	3.1980	0.0331	0.0165			
92-08276-N-3 f/LIL	2.01E-04	20			
92-08280-N-1 f/amp	7.81E-010	7	0.016	96.5344	3.3470	0.0458	0.0369			
92-08280-N-2 f/amp	3.40E-010	7	0.005	96.7358	3.2197	0.0334	0.0041			
92-08282-N-1 f/amp	2.08E-01	7	0.004	96.7470	3.0608	0.1275	0.0524			
92-08282-N-2 f/amp	4.30E-01	7	0.004	96.7609	3.1603	0.0847	0.0861			
92-08286-N-1 f/amp	2.09E-01	8	0.017	96.4481	3.4004	0.0460	0.0501			
92-08286-N-2 f/amp	5.27E-01	7	0.004	96.6516	3.2866	0.0401	0.0172			
BLANK (U/L/ml)	2.3 E-04									
STANDARD (X RECOVERY)	96									

A = acid, f = fusion, M = water leach, amp = sample, dp = duplicate, blk = blank
.. = not analyzed
average of two analyses



Battelle

Pacific Northwest Laboratories

Project Number _____

Internal Distribution

L. R. Greenwood
File/L9

Date October 9, 1992

To Susan McKinley

From J. H. Kaye *J. H. Kaye*Subject Analytical Results for Ni-59 and Ni-63 in Tank
T111, Cores 31 and 33

Nine core composite samples were received from the hot cell for ⁵⁹Ni analysis (92-08278-H1, -H2, -H3; 92-08280-H1, -H2; 92-08282-H1, -H2; and 92-08284-H1 and -H2). These represent fused composite samples and duplicates, and a fused hot cell blank from Tank T111, Cores 31 and 33. These samples were analyzed with their corresponding QC samples (two reagent blanks, a blank spike, a matrix spike, and a sample duplicate) according to procedures PNL-ALO-473, Revision-1, Nickel Separation from Radioactive Solutions, PNL-ALO-464, Procedure for Gamma Counting and Data Reduction in the Low-Level Counting Room, and PNL-ALO-474, Liquid Scintillation Counting.

Revision 1 of PNL-ALO-473 has been submitted to the ALO office. In this revised procedure, the option of 3 dimethylglyoxime precipitations was used. This was followed by anion exchange and the remainder of the procedure (steps 5.1 through 5.5, then steps 5.13 to end).

A summary of the results obtained is given in the attached table.

Chemical yields are determined by ICP measurement of stable Ni on an aliquot of the final solution prepared for LSC. The yield based on gravimetric means is determined but is not used for calculation of results. It is performed to determine if electroplating was successful. The error in the ICP measurements is quoted at $\pm 15\%$ relative; however $\pm 5\%$ is a better estimate of the error based on typical performance criteria with ICP analysis on ideal matrices such as presented with the samples submitted for analysis (2 volume percent nitric acid and no interfering ions). The data are therefore calculated with the error of $\pm 5\%$ applied to the ICP determination.

The fusion blank is used to monitor contamination due to hot cell operations; the reagent blanks are used to monitor contamination due to the laboratory analysis. The fusion blank and reagent blanks are calculated similarly to the composite fusion samples in order to provide a value which can be compared to the samples. The fusion blank gave a slightly positive result for ⁵⁹Ni, but this is insignificant compared to the sample results. A mean sample size of 0.3006 grams was used for the reagent blanks. A value of 60 ml was used for the sample volume analyzed for the reagent blanks, as this amount was used for seven out of the eleven sample volumes.

Detection limit estimates for the reagent blanks were approximately $4\text{E}-06$ for both ⁵⁹Ni and ⁶³Ni.



Project Number _____

Internal Distribution

File/LB

Date September 30, 1992

To SG McKinley

From CO Harvey *CO Harvey*

Subject Radiochemical Results for T111 (Core 31 & 33)

The radiochemical analyses were performed on four core composites, two each from Core 31 and 33. The solid core composite was prepared by potassium hydroxide (KOH) fusion, following procedure PNL-ALO-103. Duplicate samples and a single process blank were analyzed. The samples were analyzed for Total Alpha Plutonium, Uranium and for isotopic composition of the Pu and U. There was generally good agreement between duplicates for Uranium except for samples 92-08290-H1 and H2. However, the results were verified by re-analysis. The agreement of duplicates for Pu was poor, differing by a factor of approximately two. The duplicates for samples 92-08278 and 92-08290 were reanalyzed with good agreement with the original measurement, indicating acceptable analytical performance. The results of the individual analysis are included with the data. The average of the analyses is reported in the summary tables. The reason for the disagreement is unknown.

The Nickel-63 analysis are not complete and will be reported at a later date.

Concur: *[Signature]* 9-30-92

Table 13b: SST Core 31 & 33, Radiochemistry Data
 U-1aver, U MS Isotopic

Sample ID	U-1aver μg/g	U-1aver % ±	U MS Isotopic Mass Percent
Core 31 & 33 1m ± 111	3.50	3	0.0045 0.6690 0.0062 99.3195
92-02270-N-1 1/comp	4.39	3	0.0122 0.6011 0.0115 99.2952
92-02270-N-1 1/alk	4.39	3	0.0122 0.6011 0.0115 99.2952
92-02280-N-1 1/alk	7.020	4	0.0072 0.6750 0.0071 99.3097
92-02280-N-1 1/comp	3.308	4	0.0062 0.6762 0.0077 99.3099
92-02280-N-1 1/comp	4.76	4	0.0062 0.6017 0.0070 99.3051
92-02282-N-1 1/comp	4.16	4	0.0053 0.6705 0.0046 99.3198
92-02284-N-1 1/comp	3.30	3	0.0050 0.6686 0.0059 99.3219
92-02284-N-1 1/comp	3.67	4	0.0059 0.6770 0.0067 99.3103
BLANK (IC/μg)	4.8-07		
STANDARD (± RECOVERY)	95.0		

A = acid, f = fusion, W = water leach, comp = sample, dup = duplicate, blk = blank
 ** = not analyzed
 # Average of two analyses

WMC-EP-0806
 WMC-SD-WM-DP-024
 ADDENDUM 5 REV 0A

Nickel-63 and Nickel-59 Results, Tank T-111, Cores 31 and 33

ALO Sample ID	Sample Type	Ni-63 (microCi/g)	+/- one sigma*	RPD	Ni-59 (microCi/g)	+/- one sigma**	RPD
92-08278-111	Composite 1 fusion, Core 31	7.34E-03	4.0E-04		7.16E-05	1.8E-05	
92-08278-112	Composite 1 fusion, Core 31	1.13E-02	8.2E-04		9.43E-05	2.7E-05	
92-08278-113	Fusion Blank	6.17E-08	1.4E-08		<2E-08		
92-08280-111	Composite 2 fusion, Core 31	7.32E-03	4.0E-04		6.53E-05	1.8E-05	
92-08280-112	Composite 2 fusion, Core 31	3.62E-03	2.0E-04		3.30E-05	1.0E-05	
92-08280-112	Composite 2 fusion, Core 31 (duplicate)	3.53E-03	1.9E-04	3	3.36E-05	9.4E-06	2
92-08282-111	Composite 1 fusion, Core 33	6.36E-03	3.5E-04		4.74E-05	1.5E-05	
92-08282-112	Composite 1 fusion, Core 33	4.54E-03	2.5E-04		4.14E-05	1.2E-05	
92-08284-111	Composite 2 fusion, Core 33	4.26E-03	2.3E-04		3.76E-05	1.0E-05	
92-08284-112	Composite 2 fusion, Core 33	4.92E-03	2.7E-04		4.37E-05	1.2E-05	
	Reagent Blank 1**	<5E-06			<2E-06		
	Reagent Blank 2**	<6E-06			<5E-06		
		Added (dpm)	+/- one sigma*		Recovered (dpm)	+/- one sigma*	Recovery (percent)
	Blank Spike (Ni-63)	776	12		735	40	85
	Matrix Spike (Ni-63)	776	12		704	77	91

* One sigma uncertainties are based on propagation of mass, volume, counting, and yield determination uncertainties.

** Two reagent blanks were used for this batch of analyses. The results have been normalized relative to the sample volumes utilized.

Entered By J. St. George Date 10/9/92
Reviewed By Robert J. Miller Date 10-9-92

ORGANIC COMPOUND ANALYSIS REPORT
SEMIVOLATILE COMPOUNDSSAMPLE ANALYSIS REPORTED

Analysis of SST samples from Cores 31 and 33 Composites, Building 222S Hot Cell Blanks, and Field Blanks for semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS) is the subject of this report.

SAMPLE DESCRIPTION AND PREPARATION

<u>Sample ID</u>	<u>ACL Lab Number</u>
Core 31 Composite 1	92-08277
Core 31 Composite 2	92-08279
Core 33 Composite 1	92-08281
Core 33 Composite 2	92-08283
222S Hot Cell Blank	92-08285
Field Blank	92-08286

The samples were received on 07/30/92. Extractions of both samples and spiked samples were performed according to the following procedures.

- Extraction procedure PNL-ALO-120 for the solids
PNL-ALO-122 for the water sample
partitioning
- Extraction location Analytical Chemistry Hot Cells and labs
306/308, 325 building
- Extraction type Sonication, medium level soil for core
samples
Separatory funnel, medium level water for
222S Hot Cell Blank and Field Blank
samples
- Sample/Extract storage 4°C (+/- 2°)
temperature

The procedure used to extract the matrix spiked core sample resulted in a two phase extract concentrate. The second phase was eliminated by adding approximately 20 mL methylene chloride to the extract concentrates, and mixing each of these solutions with 20 grams sodium sulfate for about 30 minutes.

ANALYSIS METHOD

- GC/MS procedure: PNL-ALO-345.
- GC/MS instrumentation: HP-5890/5970 GC/MS (WB38473)
- GC/MS location: Lab 325, 325 building.

T-111 Core 31 Direct Segment Analyses

Segment 1

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		N Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18-309						
	DSC	EXOTHER	NA	EXOTHERM	EXOTHERM	NA	NA
	18-309						
	TOA	100.7	NA	87.0	86.9	87.0	0.12
	18-309						
WATER		99.2	NA	80.8	79.6	80.3	1.23

Segment 2

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		N Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18-310						
	DSC	EXOTHER	NA	EXOTHERM	EXOTHERM	NA	NA
	18-310						
	TOA	100.7	NA	87.0	NA	NA	NA
	18-310						
WATER		99.2	NA	82.4	82.6	82.5	84.61

Segment 3

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		N Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18-311						
	DSC	EXOTHER	NA	EXOTHERM	EXOTHERM	NA	NA
	18-311						
	TOA	100.7	NA	85.0	NA	NA	NA
	18-311						
WATER		99.2	NA	82.2	87.3	89.8	16.82

T-111 Core 31 Direct Segment Analyses

Segment 4

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
	10.312						
	DSC	EXOTHER	NA	EXOTHERM J		NA	NA
	10.312						
	TGA	100.7	NA	82.6	NA	NA	NA
	10.312						
S WATER		99.2	NA	J 77.3	39.6	68.5	23.84

Segment 5

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
	10.313						
	DSC	EXOTHER	NA	NO EXO	NO EXO	NA	NA
	10.313						
	TGA	99.2	NA	88.0	NA	NA	NA
	10.313						
S WATER		99.2	NA	78.4	88.4	83.4	11.99

Segment 7

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
	10.316						
	DSC	EXOTHER	NA	NO EXO	NO EXO	NA	NA
	10.316						
	TGA	99.3	NA	83.1	84.4	84.8	0.83
	10.316						
S WATER		100.7	NA	76.4	77.2	76.8	1.04

T-111 Core 31 Direct Segment Analyses

Segment 8

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		SI Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18 307						
	DSC	EXOTHER	NA	NO EXOTHER	NA	NA	NA
	18 307						
	TGA	99.3	NA	85.6	NA	NA	NA
	18 307						
% WATER		100.7	NA	76.7	76.4	76.6	0.39

Segment 9

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		SI Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18 310						
	DSC	EXOTHER	NA	NO EXO	NA	NA	NA
	18 310						
	TGA	99.3	NA	71.0	NA	NA	NA
	18 310						
% WATER		101.0	NA	76.0	74.7	75.4	2.90

Segment 9B

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
		SI Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%
	18 484						
	DSC	EXOTHER	NA	NO EXO	NO EXO	NA	NA
	18 484						
	TGA	97.8	NA	72.1	72.0	72.1	0.14
	18 484						
% WATER		99.9	NA	69.3	71.2	70.4	2.42

Special Analysis of Segment 9 (white layer)

ID #	Analyte	IMCS Std		PHEP DLE	Result #1		Result #2		Average	RPD	Std add	Spike	DST LUM	Ratio
		add (ug/L)	S Rec		(ug/g)	(ug/g)	(ug/g)	S						
ICP-Acid	Al	3000	116.60	1.24E-01	J 1.48E-02	1.51E-02	1.50E-02	3.29	99.88	65.60	2.40E+00	62.41		
	Si	3000	97.90	3.11E-01	UT -5.87E+00	-6.34E+00	-6.21E+00	-11.17	99.88	80.60	1.77E+01	-0.33		
	As	3000	90.60	3.00E+00	UT -4.23E+00	-3.55E+00	-3.89E+00	-34.40	99.88	77.60	3.00E+00	-1.96		
	Ba	10000	64.30	3.00E-01	J 5.71E-02	5.83E-02	5.68E-02	8.98	199.76	80.30	3.00E-01	1892.47		
	Ba	10000	91.40	1.00E-01	UT -1.27E-01	-7.44E-02	-1.01E-01	-51.76	199.76	88.80	1.00E-01	-1.01		
	Ca	10000	93.00	4.00E-01	J 2.38E+00	2.47E+00	2.52E+00	4.24	199.76	89.60	4.00E-01	6.31		
	Ca	10000	128.00	9.60E+01	J 9.95E-02	9.29E-02	9.12E-02	3.69	199.76	55.60	4.40E+00	207.31		
	Cr	3000	98.70	9.00E-01	J 1.97E-03	1.99E-03	1.98E-03	0.74	99.88	34.80	9.00E-01	3201.64		
	Co	10000	95.10	1.82E+00	UT 3.91E+00	3.43E+00	3.67E+00	13.16	199.76	80.30	8.00E-01	4.58		
	Co	3000	94.90	8.04E-01	J 9.27E+00	9.03E+00	9.15E+00	2.43	99.88	89.50	4.00E+01	22.97		
	Pb	3000	131.20	3.84E+00	J 1.52E+04	1.54E+04	1.53E+04	1.47	99.88	-203.0	1.00E+00	13274.6		
	Pb	3000	90.20	6.20E+00	J 9.84E+01	9.83E+01	9.83E+01	0.11	99.88	84.70	6.20E+00	15.68		
	Mg	3000	101.60	1.07E-01	J 2.25E-02	2.35E-02	2.30E-02	4.20	99.88	61.50	3.00E-01	767.33		
	Mn	3000	92.20	2.00E-01	J 4.99E-03	3.04E-03	3.01E-03	1.80	99.88	-49	2.00E-01	23066		
	Ni	3000	98.20	1.70E+00	J 9.92E-01	9.04E-01	9.99E-01	1.51	99.88	89.50	1.70E+00	32.89		
	K	3000	94.60	1.12E-01	J 3.93E-02	4.02E-02	3.93E-02	4.71	99.88	99.10	1.12E-01	35.04		
	Sc	3000	98.20	7.60E+00	J 4.60E-01	-3.74E-01	-7.17E-01	-39.92	99.88	13.90	7.60E+00	-9.43		
	Ag	3000	48.00	3.00E-01	UT 1.14E+00	9.28E-01	1.01E+00	20.15	99.88	51.10	3.00E-01	2.04		
	Na	10000	137.00	6.20E-01	J 4.91E-04	4.04E-04	4.92E-04	0.78	199.76	-346.1	3.10E+00	12971.25		
	V	10000	94.90	3.00E-01	UT 1.38E+00	1.32E+00	1.43E+00	9.44	199.76	92.20	3.00E-01	2.90		
	Zn	10000	94.80	2.03E+00	J 2.10E-01	2.04E-01	2.07E-01	3.09	199.76	90.70	3.00E-01	69.04		
	Bi	3000	66.20	7.50E+00	J 2.59E-04	2.60E-04	2.60E-04	0.24	99.88	-840.9	7.50E+00	3461.71		
	B	3000	143.60	3.64E+00	J 3.86E-01	3.28E-01	3.07E-01	13.45	99.88	47.00	6.00E-01	31.13		
	Ce	3000	106.00	1.01E-01	J 6.19E-01	6.09E-01	6.24E-01	4.76	99.88	92.60	1.01E-01	6.18		
	La	3000	98.00	1.40E+00	J 4.34E-03	4.61E-03	4.58E-03	1.17	99.88	-13.90	1.40E+00	3273.70		
	P	10000	94.10	3.80E+00	J 1.47E-04	1.69E-04	1.68E-04	1.17	199.76	-28.90	3.80E+00	2898.47		
	Si	10000	226.80	2.17E-01	J 4.03E-02	4.18E-02	4.12E-02	3.31	199.76	596.20	1.30E+00	316.66		
	Sr	10000	94.20	1.22E+00	J 4.17E-02	4.19E-02	4.18E-02	0.42	199.76	84.40	3.00E-01	1393.70		
	S	3000	112.70	1.83E-01	J 1.47E-03	1.49E-03	1.48E-03	1.24	99.88	36.40	2.70E+00	346.98		
	Se	3000	95.20	2.50E+00	UT 3.05E+00	3.00E+00	4.02E+00	48.63	99.88	88.80	1.60E+00	2.52		
	Ti	3000	94.00	4.00E-01	J 4.48E+00	4.23E+00	4.35E+00	4.49	99.88	89.50	4.00E-01	10.88		
	Zr	10000	88.30	8.00E-01	UT 1.18E-01	-1.13E-01	-1.13E-01	-4.08	99.88	139.20	8.00E-01	-14.13		

T-111 Core 31 Direct Segment Analyses

Special Analysis of Segment 9 (white layer)

	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Spike	DET LDM	Ratio	
	10-414	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S	S Rec	(ug/g)	R/DL	
IC-Water	P	107.00	<1.00E-01	J 4.77E-03	4.24E-03	4.53E-03	11.76	119.00	1.00E-01	450.30	
	Cl	96.10	<1.00E-01	J 9.43E-02	7.33E-02	8.48E-02	22.41	96.60	1.00E-01	84.80	
	NO3	85.90	<1.00E-02	J 6.12E-04	3.48E-04	3.80E-04	11.03	111.00	1.00E-02	380.00	
	NO2	101.00	<1.00E-02	J 8.42E-02	6.36E-02	7.49E-02	24.83	99.20	1.00E-02	7.49	
	PO4	96.30	<1.00E-02	J 2.88E-04	2.62E-04	2.73E-04	9.43	99.80	1.00E-02	273.00	
	SO4	96.00	<1.00E-02	J 3.31E-03	4.72E-03	3.13E-03	15.44	109.00	1.00E-02	31.13	
	P/PO4					1.67E-00					
	S/SO4					8.65E-01					
A-33		Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Spike	DET LDM	Ratio	S CNT ERR
	10-414	S Rec	(uCi/g)	(uCi/g)	(uCi/g)	(uCi/g)	S	S Rec	(uCi/g)	R/DL	
	RA D-Puo	104.60	<6.71E-4	1.33E-01	1.83E-01	1.60E-01	17.84	114.40	6.70E-04	230.73	2.30
	10-414										
	QEA	103.00	<1.2E-5	1.03E-02	9.72E-03	1.00E-02	3.79	NA	7.50E-03	133.47	2.60
	Eu-154	NA	<2.2E-4	<2.40E-4	<2.21E-4	NA	NA	NA	2.40E-04	NA	
	Eu-155	NA	<1.32E-4	<2.16E-4	<1.89E-4	NA	NA	NA	1.19E-04	NA	
	Am-241	NA	<2.62E-4	2.80E-02	2.84E-02	2.84E-02	1.40	NA	2.28E-04	123.44	2.90
	Ce-60	101.00	<8.43E-3	<8.72E-3	<7.64E-3	NA	NA	NA	8.13E-03	NA	

Hot Cell Blank

ICP-Direct	Analyte	Spike		Field Blk		Blank #1		Blank #2		Average		Spk add		Spk sub		DET LIM		Ratio	
		add (ug/L)	S Rec	(ug/L)		(ug/L)		(ug/L)		(ug/L)	S	(ug/g)		S Rec		(ug/L)		R/DL	
A-34	Al	3000	93.80	2.40E-01	UJ	2.40E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.40E-01		1.00	
	As	3000	98.30	2.37E-01	UJ	2.37E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.37E-01		1.00	
	As	3000	101.20	2.00E-01	UJ	2.00E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-01		1.00	
	Ba	10000	101.20	2.00E-00	UJ	2.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Ba	10000	99.40	2.18E-00	UJ	2.18E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.18E-00		1.00	
	Ca	10000	103.80	4.74E-00	UJ	4.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	4.00E-00		1.00	
	Ca	10000	93.20	4.40E-01	J	2.00E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	4.40E-01		1.00	
	Ca	3000	106.10	9.00E-00	UJ	9.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	9.00E-00		1.00	
	Ca	10000	104.40	8.00E-00	UJ	8.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	8.00E-00		1.00	
	Co	3000	100.40	4.00E-00	UJ	4.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	4.00E-00		1.00	
	Co	3000	103.80	1.00E-01	J	1.24E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.00E-01		1.00	
	Co	3000	104.10	6.20E-01	UJ	6.20E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	6.20E-01		1.00	
	Cr	3000	103.10	2.00E-00	J	1.01E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Cr	3000	100.30	2.00E-00	J	4.18E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Cr	3000	103.70	1.70E-01	UJ	1.70E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.70E-01		1.00	
	Fe	3000	109.60	1.12E-01	UJ	1.12E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.12E-01		1.00	
	Fe	3000	101.20	2.60E-01	UJ	2.60E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.60E-01		1.00	
	Fe	3000	88.80	2.00E-00	UJ	2.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Fe	10000	99.20	2.10E-01	J	4.91E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.10E-01		1.00	
	V	10000	101.80	2.00E-00	UJ	2.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Zn	10000	104.60	2.00E-00	J	2.62E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	Bi	3000	106.30	2.50E-01	UJ	2.50E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.50E-01		1.00	
A-34	B	3000	81.00	6.00E-00	J	1.44E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	6.00E-00		240.00	
	Ca	3000	103.10	1.01E-01	UJ	1.01E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.01E-01		1.00	
	Ca	3000	101.70	1.40E-01	UJ	1.40E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.40E-01		1.00	
	P	10000	82.40	2.80E-01	UJ	2.80E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.80E-01		1.00	
	Si	10000	73.20	2.27E-01	J	4.73E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.27E-01		263.33	
	Si	---	---	2.00E-00	UJ	1.88E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.00E-00		1.00	
	S	3000	99.40	2.70E-01	J	2.40E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	2.70E-01		1.00	
	Se	3000	103.80	1.80E-01	UJ	1.80E-01	6000	NA	NA	NA	NA	NA	NA	NA	NA	1.80E-01		1.00	
	Ti	3000	99.80	4.00E-00	UJ	4.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	4.00E-00		1.00	
	Zr	10000	83.80	8.00E-00	UJ	8.00E-00	6000	NA	NA	NA	NA	NA	NA	NA	NA	8.00E-00		1.00	

HC-30-UM

WIC-EP-0806

ndum 2, Rev. 0

T-111 Hot Cell Blank and Field Blank

Hot Cell Blank

	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Sph Rec	DET LDM	Ratio	
	18-517	S Rec	(ug/ml)	(ug/ml)	(ug/ml)	(ug/ml)	S	S	(ug/ml)	R/DL	
HYAA	As	90.60	< 3.0E-4	UJ < 3.0E-3	< 3.0E-3	NA	NA	NA	< 3.0E-3	NA	
	Sa	100.00	< 3.0E-4	UJ < 3.0E-3	< 3.0E-3	NA	NA	NA	< 3.0E-3	NA	
CYAA	Hg	90.00	< 3.0E-4	UJ < 3.0E-3	< 3.0E-3	NA	NA	NA	< 3.0E-3	NA	
	18-504										
IC-Water	P	100.00	< 1	UJ < 1	< 1	NA	NA	100	< 1	NA	
	Cl	100.00	< 1	UJ < 1	< 1	NA	NA	120	< 1	NA	
	NO3	94.60	< 1.0	UJ < 1.0	< 1.0	NA	NA	85.9	< 1.0	NA	
	NO2	100.00	< 1.0	UJ < 1.0	< 1.0	NA	NA	99.7	< 1.0	NA	
	PO4	90.70	< 1.0	UJ < 1.0	< 1.0	NA	NA	97.3	< 1.0	NA	
	SO4	92.80	< 1.0	UJ < 1.0	< 1.0	NA	NA	95.7	< 1.0	NA	
	18-513										
TOC-Water	TOC	90.70	3 uOC	UJ < 3	< 3	NA	NA	NA	300	NA	
	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Sph Rec	DET LDM	Ratio	Relative
	18-496	S Rec	(uCi/mL)	(uCi/mL)	(uCi/mL)	(uCi/mL)	S	S	(ug/mL)	R/DL	Count Err
RAD-Water	TA	100.90	< 4.39E-6	< 3.13E-6	< 4.39E-6	NA	NA	NA	4.39E-04	NA	1.00E-01
	18-496										
	TB	100.10	9.38E-5	< 7.23E-5	< 7.94E-5	NA	NA	NA	9.38E-05	NA	6.60E-00
	18-496										
GEA	CS-137	100.00	< 1.17E-6	< 1.19E-6	< 1.20E-6	NA	NA	NA	1.17E-04	NA	NA
	Eu-154	N/A	< 2.97E-6	< 3.07E-6	< 3.96E-6	NA	NA	NA	2.97E-04	NA	NA
	Eu-155	N/A	< 1.81E-6	< 1.82E-6	< 1.77E-6	NA	NA	NA	1.82E-04	NA	NA
	Am-241	N/A	< 3.34E-6	< 3.38E-6	< 4.41E-6	NA	NA	NA	3.34E-04	NA	NA
	Co-60	100.00	< 1.04E-6	< 1.15E-6	< 1.15E-6	NA	NA	NA	1.04E-04	NA	NA

WHC-EP-0806

WHC-SD-WM-DP-024 ADDENDUM 2, REV 0

ICF-A-14	1001	Analyte	LMCS 14	Blank	Field Blank	Blank 01	Blank 02	Average	RPD	SP-14	SP-14	SP-14	DET L41	Ratio
			Ad (g/L)	Blank	Blank	(g/L)	(g/L)	(g/L)	Blank	(g/L)	(g/L)	(g/L)	(g/L)	R/DL
Al	3000		91.80	3.40E-01	J	1.17E-02	1.14E-02	3.42E-02	3.04	NA	NA	NA	3.40E-01	1.22
Se	3000		98.30	-3.37E-02	WJ	1.77E-02	1.77E-02	1.77E-02	8.00	NA	NA	NA	1.77E-02	1.00
As	3000		101.20	3.00E-01	WJ	3.00E-01	3.00E-01	3.00E-01	8.00	NA	NA	NA	3.00E-01	1.00
Ba	10000		101.20	3.00E-01	WJ	6.62E-00	4.49E-00	3.54E-00	35.31	NA	NA	NA	3.00E-00	1.11
Ba	10000		99.40	3.11E-00	WJ	1.00E-00	1.00E-00	1.00E-00	8.00	NA	NA	NA	1.00E-00	1.00
Ca	10000		105.80	4.74E-00	WJ	4.00E-00	4.57E-00	4.28E-00	12.89	NA	NA	NA	4.00E-00	1.87
Ca	10000		108.10	4.40E-01	WJ	3.00E-00	3.00E-00	3.00E-00	8.00	NA	NA	NA	4.00E-01	1.44
Co	10000		104.40	8.00E-00	WJ	8.00E-00	8.00E-00	8.00E-00	8.00	NA	NA	NA	8.00E-00	1.44
Co	3000		100.40	4.00E-00	WJ	1.32E-01	8.79E-00	1.10E-01	48.81	NA	NA	NA	4.00E-00	1.00
Fe	3000		103.80	1.00E-01	WJ	1.00E-01	1.00E-01	1.00E-01	8.00	NA	NA	NA	1.00E-01	1.00
Fe	3000		104.10	6.20E-01	WJ	6.20E-01	6.20E-01	6.20E-01	8.00	NA	NA	NA	6.20E-01	1.00
Mg	3000		103.10	3.00E-00	J	3.33E-01	3.62E-01	3.69E-01	11.38	NA	NA	NA	3.00E-00	1.00
Mn	3000		100.30	3.00E-00	J	3.07E-01	4.80E-01	4.94E-01	8.28	NA	NA	NA	3.00E-00	1.00
Ni	3000		105.20	1.20E-01	WJ	1.20E-01	1.20E-01	1.20E-01	8.00	NA	NA	NA	1.20E-01	1.00
K	3000		109.80	1.12E-02	WJ	1.12E-02	1.12E-02	1.12E-02	8.00	NA	NA	NA	1.12E-02	1.00
Na	3000		101.20	7.60E-01	WJ	7.60E-01	7.60E-01	7.60E-01	8.00	NA	NA	NA	7.60E-01	1.00
Ag	3000		88.30	3.00E-00	WJ	3.00E-00	3.00E-00	3.00E-00	8.00	NA	NA	NA	3.00E-00	1.00
Na	10000		89.20	3.10E-01	J	8.20E-02	7.94E-02	8.07E-02	3.25	NA	NA	NA	3.10E-01	1.00
V	10000		101.80	3.00E-00	WJ	3.00E-00	3.00E-00	3.00E-00	8.00	NA	NA	NA	3.00E-00	1.00
Zn	10000		104.60	3.00E-00	J	4.22E-01	4.12E-01	4.27E-01	4.44	NA	NA	NA	3.00E-00	1.00
Bi	3000		104.30	3.30E-01	WJ	3.30E-01	3.30E-01	3.30E-01	8.00	NA	NA	NA	3.30E-01	1.00
B	3000		88.00	6.00E-00	J	1.79E-02	1.33E-02	1.76E-02	3.21	NA	NA	NA	6.00E-00	281.88
Co	3000		105.10	1.01E-01	WJ	1.01E-02	1.01E-02	1.01E-02	8.00	NA	NA	NA	1.01E-02	1.00
La	3000		102.20	1.40E-01	WJ	3.42E-02	2.16E-01	2.27E-01	11.78	NA	NA	NA	1.40E-01	1.00
P	10000		82.40	3.80E-01	J	8.18E-02	8.10E-02	8.14E-02	1.04	NA	NA	NA	3.80E-01	14.83
Si	10000		78.20	2.27E-01	J	4.61E-02	4.47E-02	4.54E-02	3.16	NA	NA	NA	2.27E-01	449.44
Br	3000			3.00E-00	J	2.12E-01	3.18E-01	3.62E-01	3.48	NA	NA	NA	3.00E-00	12.16
S	3000		89.40	3.20E-01	J	2.19E-02	2.41E-02	2.34E-02	4.30	NA	NA	NA	3.20E-01	9.40
Se	3000		102.80	1.60E-01	WJ	1.60E-02	1.60E-02	1.60E-02	8.00	NA	NA	NA	1.60E-01	1.00
Si	3000		89.20	4.00E-00	WJ	4.00E-00	4.00E-00	4.00E-00	8.00	NA	NA	NA	4.00E-00	1.00
Zr	3000			8.00E-00	WJ	8.00E-00	8.00E-00	8.00E-00	8.00	NA	NA	NA	8.00E-00	1.00

T-111 Hot Cell Blank and Field Blank

Field Blank

	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Sgt Rec	DET LIM	Ratio	
	18.516	18 Rec	(ug/ml)	(ug/ml)	(ug/ml)	(ug/ml)	%	%	(ug/ml)	R/DL	
HYAA	As	98.60	<3.0E-4	UJ <3.0E-3	<3.0E-3	NA	NA	NA	<3.0E-3	NA	
	Sa	100.00	<3.0E-4	UJ <3.0E-3	<3.0E-3	NA	NA	NA	<3.0E-3	NA	
CYAA	Hg	93.00	<3.0E-4	UJ <3.0E-3	<3.0E-3	NA	NA	NA	<3.0E-3	NA	
	18.503										
IC-Water	F	101.00	<1	UJ 3.30E-01	3.30E-01	3.30E-01	3.00	3.00E-03	<1	NA	
	Cl	103.00	<1	UJ <1	<1	NA	NA	3.30E-03	<1	NA	
	NO3	94.60	<1.0	UJ 4.30E-00	4.30E-00	NA	NA	6.39E-01	<1.0	NA	
	NO2	101.00	<1.0	UJ <1.0	<1.0	NA	NA	9.97E-01	<1.0	NA	
	PO4	93.70	<1.0	UJ 2.34E-00	2.43E-00	2.30E-00	3.61	9.73E-01	<1.0	NA	
	SO4	92.90	<1.0	UJ <1.0	<1.0	NA	NA	9.37E-01	<1.0	NA	
	18.512										
TOC-Water		98.70	3.00 C	UJ <3.3	<3.3	NA	NA	NA	300	NA	
A-37	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Sgt Rec	DET LIM	Ratio	Relative
	18.497	18 Rec	(uCi/ml)	(uCi/ml)	(uCi/ml)	(uCi/ml)	%	%	(uCi/ml)	R/DL	Count Eff
RAD-Water	TA	100.90	<4.39E-6	<4.39E-6	<4.39E-6	NA	NA	NA	4.39E-04	NA	70.70
	18.497										
	TB	100.10	<9.30E-3	1.16E-04	1.20E-04	1.18E-04	3.30	NA	9.30E-03	1.26	3.50
	18.497										
UEA	Cs-137	101.00	<1.17E-6	1.32E-03	1.66E-03	1.39E-03	2.50	NA	1.17E-04	47.78	3.40
	Eu-154	NA	<1.87E-6	<1.81E-6	<1.89E-6	NA	NA	NA	1.87E-04	NA	NA
	Eu-155	NA	<1.81E-6	<1.93E-6	<1.84E-6	NA	NA	NA	1.81E-04	NA	NA
	Am-241	NA	<1.34E-6	<1.72E-6	<1.71E-6	NA	NA	NA	1.34E-04	NA	NA
	Ce-60	101.00	<1.04E-6	<1.07E-6	<1.11E-6	NA	NA	NA	1.04E-04	NA	NA

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MHC-SD-WM-DP-024 ADDENDUM 2, REV 0

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T-111 Core 31 Composite 1

El. #	Analyte	LMCS Bal		PREP BLK	Result #1	Result #2	Average	R/D	Spt add	Spts	DET LBL	Ratio	mcg	MnI
		val (ug/l)	S Rec	ug/g	(ug/g)	(ug/g)	(ug/g)	S	(ug/g)	S Rec	(ug/g)	R/DL	conc	ug/g
10.449	ICP-Acid													
	Al	1000	121.40	1.21E-01	J 3.38E-02	3.00E-02	3.04E-02	1.41	33.94	43.40	2.40E-00	243.33		
	As	1000	96.90	1.77E-01	UT 2.19E-01	1.90E-01	3.04E-01	38.03	33.94	106.60	1.77E-01	1.72		
	As	1000	87.90	3.00E-00	UT 3.17E-00	3.13E-00	3.13E-00	1.47	33.94	67.00	3.00E-00	1.03		
	Ba	2000	89.90	3.99E-01	J 3.73E-01	3.64E-01	3.70E-01	1.33	107.87	91.30	3.00E-01	190.14		
	Ba	2000	84.40	1.00E-01	UT 1.04E-01	1.04E-01	1.03E-01	1.43	107.87	84.40	1.00E-01	1.03		
	Ca	2000	87.90	4.44E-01	UT 7.23E-00	7.19E-00	7.22E-00	0.74	107.87	88.90	4.00E-01	18.03		
	Ca	2000	139.00	6.12E-01	J 3.26E-03	2.13E-03	2.20E-03	6.04	107.87	-48.40	4.40E-00	498.96	0.11	0.1
	Cr	1000	93.30	9.00E-01	J 1.89E-03	1.83E-03	1.84E-03	3.23	33.94	0.18	9.00E-01	2044.98		0.1
	Co	2000	89.18	8.00E-01	UT 3.38E-00	3.42E-00	3.40E-00	1.91	107.87	89.60	8.00E-01	4.23		
	Cu	1000	84.18	4.00E-01	J 2.37E-01	2.48E-01	2.32E-01	3.43	33.94	89.30	4.00E-01	63.33		
	Pb	1000	93.60	6.37E-00	J 1.83E-04	1.89E-04	1.82E-04	3.18	33.94	-680.6	1.00E-00	19316.3		
	Pb	1000	89.18	6.20E-00	J 4.81E-02	4.68E-02	4.73E-02	2.32	33.94	70.30	6.20E-00	76.34		
	Mg	1000	97.30	3.18E-00	J 4.31E-02	4.32E-02	4.33E-02	1.37	33.94	43.30	3.00E-01	1430.74		
	Mn	1000	84.60	2.00E-01	J 6.31E-03	6.07E-03	6.19E-03	3.78	33.94	-234.3	2.00E-01	30937.1		
	Ni	1000	89.70	1.70E-00	J 1.34E-02	1.49E-02	1.31E-02	1.61	33.94	84.30	1.70E-00	89.41		
	K	1000	103.80	1.67E-01	J 1.11E-03	1.01E-03	1.10E-03	2.24	33.94	38.70	1.12E-01	97.87		
	Se	1000	80.80	7.60E-00	UT 8.04E-00	7.92E-00	7.98E-00	1.47	33.94	8.00	7.60E-00	1.03		
	Ag	1000	93.90	3.00E-01	J 2.03E-02	2.02E-02	2.03E-02	0.81	33.94	87.90	3.00E-01	403.28		
	Na	2000	133.00	1.10E-02	J 3.80E-04	3.71E-04	3.76E-04	2.42	107.87	-331.3	3.10E-00	12118.9		
	V	2000	89.60	3.31E-01	J 1.31E-01	1.23E-01	1.27E-01	3.62	107.87	90.40	3.00E-01	23.39		
	Zn	2000	88.20	9.18E-01	J 8.20E-01	7.68E-01	7.94E-01	6.37	107.87	88.30	3.00E-01	264.80		
	Bi	1000	34.40	7.50E-00	J 2.37E-04	2.34E-04	2.34E-04	1.37	33.94	-319.0	7.30E-00	3161.34		
	B	1000	813.18	2.76E-01	UT 3.06E-01	2.34E-01	2.71E-01	27.87	33.94	23.00	6.00E-01	43.28		
	Co	1000	107.80	1.81E-01	UT 3.13E-01	3.39E-01	3.26E-01	7.92	33.94	97.70	1.81E-01	3.23		
	La	1000	93.30	1.40E-00	J 3.78E-03	3.64E-03	3.72E-03	3.27	33.94	-38.40	1.40E-00	2457.03	0.08	0.0
	P	2000	87.60	9.38E-00	J 1.00E-04	1.02E-04	1.01E-04	1.90	107.87	493.40	3.80E-00	1718.84	0.93	0.0
	Si	2000	203.00	8.08E-01	J 4.31E-02	3.29E-02	4.03E-02	19.26	107.87	8.40	1.30E-00	370.84		
	Si	2000	89.40	1.00E-01	J 2.83E-02	2.78E-02	2.82E-02	2.42	107.87	81.80	1.00E-01	938.96		
	S	1000	100.40	9.39E-00	J 1.24E-03	1.31E-03	1.33E-03	2.23	33.94	91.80	2.70E-00	654.66	0.11	0.1
	Se	1000	84.30	3.64E-00	UT 4.13E-00	4.38E-00	4.21E-00	3.33	33.94	89.40	1.60E-00	2.63		
	Ti	1000	89.80	4.00E-01	J 2.93E-01	2.94E-01	2.94E-01	1.08	33.94	83.30	4.00E-01	73.38		
	Zr	2000	88.18	8.00E-01	UT 8.47E-01	8.34E-01	8.40E-01	1.48	107.87	8.60	--	NA		

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ADDENDUM 2, REV 2

T-111 Core 31 Composite 1

Direct/Unhom.	Analyte	Sid #1	PREP D1K	Recon #1	Recon #2	Average	RPD	Spt Rec	DET LDM	Bottle	msq	Met Bal
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S	IS	(ug/g)	B/DL	msq	(g/g)
	12.433											
	DSC	EXOTHER	NA	EXOTHERM	EXOTHERM	NA	NA	NA	NA	NA		
	1433			(S)	(S)	(S)						
	TOA	91.80	NA	85.30	61.30	79.30	32.74	NA	NA	NA		
	12.416, 417			(S)	(S)	(S)						
S WATER	SWTR	99.70	NA	74.40	74.80	74.60	0.54	NA	NA	NA		0.746
	1436											
HYAA	A ₆ —	PNL										
	B ₆ —	PNL										
CVAA	Hg	101.2	< 125	1.41E-00	1.70E-00	1.55E-00	11.81	123.00	0.125	12.72		
	12.440											
CN-DH	CN	99.80	< 3.0	< 4.0	< 4.5	NA	NA	69.10	3.0	NA	0.000	0
	CuHf-CuH6					NA						

T-111 Core 31 Composite 1

ID	Analyte	LMCS Std		PREP BLK	Result #1		Result #2	Average	RPD	Std add		DET LDM	Ratio
		added	S Rec		(ug/g)	(ug/g)				(ug/L)	S Rec		
10 437				(ug/g)				(ug/g)	S			(ug/g)	R/DL
ICP-Water	Al	3000	93.60	2.40E-00	UJ 6.43E-00	6.99E-00	6.71E-00	6.33		1230	99.30	2.40E-00	2.80
	Sb	3000	98.40	1.77E-01	UJ 1.77E-01	1.77E-01	1.77E-01	0.10		1230	74.10	1.77E-01	1.00
	As	3000	93.60	3.00E-00	UJ 3.00E-00	3.00E-00	3.00E-00	0.10		2300	44.20	3.00E-00	1.00
	Ba	10000	98.30	3.00E-01	UJ 3.00E-01	3.09E-01	3.03E-01	3.13		2300	98.80	3.00E-01	1.02
	Be	10000	96.90	1.00E-01	UJ 9.99E-02	1.00E-01	1.00E-01	0.10		2300	98.60	1.00E-01	1.00
	Cd	10000	97.90	4.00E-01	UJ 4.00E-01	4.00E-01	4.00E-01	0.10		2300	97.20	4.00E-01	1.00
	Ca	10000	90.70	2.10E-01	UJ 2.12E-01	2.03E-01	2.08E-01	1.33		2300	91.00	4.40E-00	11.55
	Cr	3000	100.60	9.00E-01	J 2.07E-02	2.11E-02	2.09E-02	2.23		1230	96.80	9.00E-01	232.21
	Co	10000	96.40	8.00E-01	UJ 7.99E-01	8.00E-01	8.00E-01	0.10		2300	97.60	8.00E-01	1.00
	Cu	3000	93.80	4.00E-01	UJ 4.00E-01	4.00E-01	4.00E-01	0.10		1230	96.90	4.00E-01	1.00
	Fe	3000	97.00	1.00E-00	J 1.02E-02	1.70E-01	7.94E-01	34.97		1230	94.70	1.00E-00	79.63
	Pb	3000	94.00	6.20E-00	UJ 6.19E-00	7.91E-00	7.03E-00	24.34		1230	93.20	6.20E-00	1.14
	Mg	3000	98.70	6.60E-01	UJ 3.22E-00	2.67E-00	2.94E-00	10.77		1230	99.70	3.60E-01	9.81
	Mn	3000	93.50	3.00E-01	J 1.93E-01	1.01E-01	1.47E-01	62.70		1230	93.70	2.60E-01	73.45
	Ni	3000	97.30	1.70E-00	UJ 1.70E-00	1.70E-00	1.70E-00	0.10		1230	98.10	1.70E-00	1.00
	K	3000	103.10	3.12E-01	J 7.28E-02	7.40E-02	7.34E-02	1.63		1230	83.10	1.12E-01	65.53
	Se	3000	93.90	7.60E-00	UJ 7.39E-00	7.60E-00	7.60E-00	0.10		1230	101.80	7.60E-00	1.00
	Ag	3000	101.00	3.00E-01	UJ 4.99E-01	7.79E-01	6.39E-01	43.78		1230	102.30	3.00E-01	1.26
	Na	10000	96.60	2.88E-01	J 3.39E-04	3.41E-04	3.40E-04	0.49		2300	-27.70	3.10E-00	10973.7
	V	10000	93.30	3.00E-01	UJ 4.99E-01	3.00E-01	3.00E-01	0.10		2300	97.60	3.00E-01	1.00
	Zn	10000	97.90	-1.49E-00	UJ 3.00E-01	3.00E-01	3.00E-01	0.10		2300	97.00	3.00E-01	1.00
	Bi	3000	99.60	7.30E-00	J 1.47E-02	8.34E-01	1.13E-02	53.30		1230	39.80	7.30E-00	11.39
	B	3000	87.80	6.00E-01	J 3.11E-00	3.30E-00	3.21E-00	11.84		1230	86.10	6.00E-01	3.51
	Co	3000	99.00	1.01E-01	UJ 1.01E-01	1.01E-01	1.01E-01	0.10		1230	99.30	1.01E-01	1.00
	La	3000	99.10	1.40E-00	J 7.03E-00	3.00E-00	6.02E-00	33.93		1230	100.30	1.40E-00	4.30
	P	10000	93.40	3.80E-00	J 3.89E-01	3.63E-01	3.76E-01	4.43		2300	82.80	3.80E-00	993.36
	Si	10000	80.00	1.30E-00	J 3.30E-03	3.63E-03	4.38E-03	42.48		2300	99.10	1.30E-00	336.53
	Br	10000	97.30	3.00E-01	UJ 1.48E-00	9.34E-01	1.21E-00	43.14		2300	99.00	3.00E-01	4.63
	S	3000	97.10	2.70E-00	J 1.18E-03	1.21E-03	1.19E-03	2.13		1230	93.70	2.70E-00	443.49
	Se	3000	92.70	1.80E-00	UJ 1.60E-00	1.60E-00	1.60E-00	0.10		1230	93.00	1.60E-00	1.00
	Ti	3000	97.80	4.00E-01	UJ 4.00E-01	4.00E-01	4.00E-01	0.10		1230	99.40	4.00E-01	1.00
	Zr	10000	98.30	3.00E-01	UJ 7.99E-01	8.00E-01	8.00E-01	0.10		--	--	--	--

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MHC-SD-WM-DP-024 ADDENDUM 2, REV 0

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T-111 Core 31 Composite 1

	Analyte	Std St	PREP BLK	Result #1	Result #2	Average	RPD	SpA Rec	DET LDM	Ratio	meq	Dist Det
		St Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	%	(ug/g)	R/DL	ions	(g/g)
IC-Water	18.437											
	IF	04.30	<1.00E-1	J 3.01E-03	3.14E-03	3.09E-03	3.57	117.00	1.00E-01	301.30	0.16	0.00
	IC	103.0	<1.00E-1	J 4.60E-03	4.73E-03	4.70E-03	1.49	119.00	1.00E-01	44.93		0.00
	IN03	93.20	<1.00E-2	J 4.41E-04	4.43E-04	4.43E-04	0.90	101.00	1.00E-03	443.00	0.71	0.04
	IN02	90.10	<1.00E-2	UJ <1.00E-3	<1.10E-3	NA	NA	104.30	1.00E-03	NA		0.00
	IC04	96.30	<1.00E-2	J 1.67E-04	1.56E-04	1.61E-04	0.81	86.00	1.00E-03	161.30		0.01
	SO4	90.70	<1.00E-2	J 3.69E-03	3.69E-03	3.69E-03	0.00	81.70	1.00E-03	36.90		0.01
	P ACID IC/IC04 IC					1.97E-00						
	S ACID IC/IC04 IC					0.97E-01						
	18.437											
Spot-Water	NO3	100.0	<1.00E-1	J 0.13E-03	0.49E-03	0.31E-03	0.63	97.30	1.00E-01	19.04		0.00
	NO3 (IC)/NO3 Spec.					44.13						
	NO3 (IC)/NO3 (SPEC)					0.40						
A-41	18.437											
	pH	100.7	NA	J 10.17	10.10	10.13	0.20	NA	NA	NA		
	18.437											
NH3-Water	NH3	97.60	<4500	UJ <4500	<4500	NA	NA	98.40	4.30E-03	NA		
	18.437											
	18.437											
TOC-Water	TOC	98.30	<650	J 3.66E-03	3.30E-03	3.49E-03	10.89	93.80	1.00E-02	6.98		
	Acetate Eq		0.72E-04								0.291	0.000
	18.437											
TIC-Water	CO3	103.3	<500	UJ 6.30E-03	<3.00E-3	NA	NA	103.30	1.00E-03	NA	0.000	0.000

WHC-SD-WM-DP-024 ADDENDUM 2, REV 0

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	Analyte	IMCS 54		PREF BLK	Result #1	Result #2	Average	RFD	Spl 544	Spl 544	DET 104	Ratio	mg	Mn
JE 446		Added	% Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	(ug/g)	% Rec	(ug/g)	R/DL	Units	G
ICP-Fusion	Al	3000	93.30	1.20E-01	J 6.56E-01	6.32E-02	6.44E-02	3.74	1250	94.20	1.20E-01	53.70		
	Sb	3000	90.70	6.82E-01	UJ 8.82E-01	8.87E-01	8.84E-01	0.20	1250	101.80	6.82E-01	1.04		
	As	3000	91.30	1.50E-01	UJ 1.50E-01	1.50E-01	1.50E-01	0.20	2500	44.80	1.50E-01	1.00		
	Ba	10000	97.00	1.50E-00	J 6.04E-01	5.72E-01	5.88E-01	3.34	2500	98.00	1.50E-00	39.21		
	Ba	10000	99.80	1.15E-00	UJ 1.00E-01	2.01E-01	2.01E-01	0.20	2500	99.70	1.00E-01	1.00		
	Ca	10000	99.40	2.00E-00	UJ 2.16E-00	2.04E-00	2.10E-00	22.11	2500	100.40	2.00E-00	4.13		
	Ca	10000	92.40	1.28E-02	J 2.93E-03	2.58E-03	2.76E-03	12.70	2500	94.80	2.20E-01	123.39		
	Cr	3000	102.30	4.50E-00	J 4.92E-03	1.84E-03	1.89E-03	2.91	1250	106.00	4.50E-00	420.02		
	Co	10000	99.10	4.00E-00	UJ 1.03E-01	9.70E-00	1.01E-01	7.00	2500	99.40	4.00E-00	2.32		
	Cu	3000	93.60	3.31E-00	J 2.39E-01	2.64E-01	2.63E-01	2.64	1250	96.30	3.00E-00	18.17		
	Fe	3000	97.80	7.13E-01	J 2.01E-04	2.01E-04	2.01E-04	2.88	1250	123.30	5.00E-00	4103.72		
	Pb	3000	91.70	3.10E-01	J 4.33E-02	4.37E-02	4.40E-02	3.76	1250	88.90	3.10E-01	14.19		
	Mg	3000	99.30	1.36E-01	J 4.32E-02	4.24E-02	4.28E-02	6.32	1250	100.90	1.50E-00	292.00		
	Mn	3000	94.60	3.04E-00	J 6.47E-03	6.29E-03	6.38E-03	2.83	1250	103.20	1.00E-00	6381.39		
	Ni	3000	98.80	5.91E-01	J 5.61E-03	5.94E-03	5.77E-03	3.64	1250	107.00	6.50E-00	679.30		
	K	3000	100.10	3.60E-01	UJ 3.60E-01	3.61E-01	3.61E-01	0.20	1250	1344.3	3.60E-01	1.00		
	Se	3000	93.20	3.80E-01	UJ 3.80E-01	3.81E-01	3.80E-01	0.20	1250	96.60	3.80E-01	1.00		
	Ag	3000	100.80	8.50E-00	J 2.18E-02	2.10E-02	2.14E-02	3.39	1250	103.10	2.50E-00	83.74		
	Na	10000	93.60	4.69E-03	J 4.01E-04	3.96E-04	3.98E-04	1.28	2500	109.80	1.53E-01	2569.99		
	V	10000	98.20	2.50E-00	J 1.32E-01	1.11E-01	1.21E-01	17.23	2500	99.90	2.50E-00	4.85		
	Zn	10000	99.60	1.36E-01	J 1.11E-02	9.71E-01	1.04E-02	13.00	2500	99.80	1.50E-00	69.22		
	Bi	3000	94.40	3.73E-01	J 2.14E-04	2.03E-04	2.09E-04	4.47	1250	79.40	3.73E-01	338.23		
	B	3000	83.10	3.00E-00	UJ 3.00E-00	3.01E-00	3.00E-00	0.20	1250	83.10	3.00E-00	1.00		
	Co	3000	101.30	3.03E-01	UJ 3.03E-01	3.04E-01	3.04E-01	0.20	1250	97.80	3.03E-01	1.00		
	La	3000	99.30	7.00E-00	J 2.73E-03	2.63E-03	2.68E-03	3.24	1250	103.40	7.00E-00	527.61		
	P	10000	101.90	2.90E-01	J 1.19E-04	1.12E-04	1.16E-04	8.67	2500	103.70	2.90E-01	398.76		
	Si	10000	75.18	6.79E-01	J 6.04E-03	5.88E-03	5.96E-03	2.75	2500	79.10	6.50E-00	917.29	0.45	
	Si	10000	97.30	1.50E-00	J 2.94E-03	2.84E-03	2.89E-03	3.29	2500	98.90	1.50E-00	202.19		
	S	3000	101.00	2.77E-01	J 1.34E-03	1.33E-03	1.33E-03	0.97	1250	102.60	1.33E-01	100.28		
	Sn	3000	97.00	9.16E-00	UJ 9.00E-00	9.02E-00	9.01E-00	0.20	1250	93.80	9.00E-00	1.00		
	Ti	3000	96.80	2.00E-00	J 7.23E-01	7.32E-01	7.29E-01	0.98	1250	97.80	2.00E-00	34.43		
	Zr	10000	93.20	4.00E-00	UJ 4.00E-00	4.01E-00	4.00E-00	0.20	1250	--	--	--		

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T-111 Core 31 Composite 1

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T-111 Core 31 Composite 1

	Analyte	Std #1	Prep Dil	Result #1	Result #2	Average	RPD	Sys Rec	DET LDM	Ratio	Det.
		# Rec	(uCi/g)	(uCi/g)	(uCi/g)	(uCi/g)	#	#	(uCi/g)	R/DL	Ct Err
	10-466										
	Am-241	100.1	<1.70E-3	J 3.01E-02	4.41E-02	4.14E-02	14.01	22.50	5.70E-03	7.26	6.10
								17.00			6.00
	Com-244			none detected		NA	NA			NA	
	Am-241 (OEAP)/Am-241 (ALPHA)					8.11E-00					
	J-8466										
	HP-237	70.30	<1.49E-2	UJ <1.24E-2	<1.21E-2	NA	NA	73.60	3.42E-03	NA	3.30
	10-466										
	IC-99	110.1	<1.41E-4	3.31E-03	4.03E-03	3.14E-03	7.39	71.00	8.50E-04	6.03	4.50
								72.00			
	10-466										
	I-129	119.1	<1.60E-2	LT <1.72E-2	<1.74E-2	NA	NA	57.40	1.72E-02	NA	
								51.10			

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T-111 Core 31 Composite 1

	Analyte	Std #1	Prep Blk	Result #1	Result #2	Average	RPD	Sgt Rec	DET LDM	Ratio	Rel.	
		S Rec	(uCVg)	(uCVg)	(uCVg)	(uCVg)	#	#	(uCVg)	M/DL	Cl Err	
	10-466											
	Se-90	88.60	< 40E-3	J 7.14E-00	6.97E-00	7.14E-00	5.17	92.00	7.10E-01	1007.7	0.30	
								91.00				
	10-457											
	H-3	85.90	< 13E-4	< 13E-4 UJ	< 13E-4	NA	NA	81.30	3.13E-04	NA	6.60	
	10-457											
	C-14	93.30	< 23E-4	< 23E-4 UJ	3.80E-04	NA	NA	93.70	2.23E-04	NA	4.30	
	10-466											
	Se-79	80.00	< 6.71E-3	UJ < 6.11E-3	< 6.05E-3	NA	NA	89.10	3.61E-03	NA	4.30	
								93.60				
A-45	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Sgt Rec	DET LDM	M/DL	ICLP LDM	Result/Lim
		S Rec	(mg/L)	(mg/L)	(mg/L)	(mg/L)	#	#	(mg/L)		(mg/L)	
	10-641											
	ICLP-ICP											
	Ag	99.40	3.00E-03	J 4.11E-02	2.30E-02	3.31E-02	48.71	2.90	3.00E-03	6.61	3.00	0.01
	Ba	97.80	3.00E-03	J 2.73E-02	1.30E-02	2.12E-02	38.16	3.40	3.00E-03	7.03	100.00	0.00
	Ca	97.00	4.00E-03	J 2.00E-02	2.00E-02	2.00E-02	0.00	94.10	4.00E-03	3.00	3.00	0.02
	Cr	103.30	9.00E-03	J 8.69E-00	8.32E-00	8.60E-00	1.97	30.30	9.00E-03	933.79	3.00	1.72
	Pb	98.30	6.70E-02	J 3.91E-01	3.11E-01	3.51E-01	22.74	6.70	6.20E-02	3.64	3.00	0.07
	As	93.30	3.00E-02	J 1.30E-01	1.30E-01	1.30E-01	0.00	--	3.00E-02	3.00	3.00	0.03
	Se	123.10	1.11E-01	J 1.21E-00	1.34E-00	1.27E-00	10.37	--	7.60E-02	16.77	1.00	1.27
	10-632											
ICLP-CVAA	11g	97.30	< 0.0100	UJ 4.30E-02	4.30E-02	4.30E-02	0.00	100.10	1.00E-02	4.30	0.02	2.23

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T-111 Core 31 Composite 2

Direct/Unburn	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD	Spl Rec	DET LBM	Ratio	meq	Mat Bal
		8 Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	8	8	(ug/g)	R/DL	tons	(g/g)
	18 434											
	DIC	EXO	NA	EXOTHERM	EXOTHERM	NA	NA	NA	NA	NA		
	18 434			(#)	(#)	(#)						
	TOA	91 80	NA	71.20	69.20	70.20	2 85	NA	NA	NA		
	18 418, 419			(#)	(#)	(#)						
R WATER	SWTR	99.70	NA	73.90	73.90	73.90	8 00	NA	NA	NA		0.739
	As	PNL										
HYAA	Se	PNL										
CVAA	Hg	101.2	< 113	1.10E-00	1.79E-00	1.43E-00	4.90	133.00	1.23E-01	ERR		
	18 441											
CN-Die	CN	99.90	< 50	0.37	0.41	NA	NA	00.10	3.00E-00	NA		
	CaNiFeCN6					NA						

ID	Analyte	INCH Std		PREP BLK	Result #1		Average	RPD	Spk add		DETLIM	Ratio
		std	Rec		(ug/g)	(ug/g)			(ug/L)	Rec		
18.439				(ug/g)			(ug/g)	%			(ug/g)	N/DL
ICP-Water	Al	3000	93.60	2.40E-00	UT 1.02E-01	1.04E-01	1.03E-01	1.70	1250	99.50	2.40E-00	4.29
	Sb	3000	93.40	1.77E-01	UT 1.77E-01	1.77E-01	1.77E-01	0.60	1250	74.10	1.77E-01	1.00
	As	3000	93.40	3.00E-00	UT 3.00E-00	3.00E-00	3.00E-00	0.60	2500	44.20	3.00E-00	1.00
	Ba	10000	93.30	3.00E-01	UT 3.32E-01	3.00E-01	4.16E-01	33.84	2500	98.80	3.00E-01	1.39
	Ba	10000	94.90	3.00E-01	UT 9.99E-02	9.99E-02	9.99E-02	0.00	2500	98.60	3.00E-01	1.00
	Ca	10000	97.90	4.00E-01	UT 4.00E-01	4.00E-01	4.00E-01	0.00	2500	97.20	4.00E-01	1.00
	Ca	10000	90.70	2.10E-01	UT 6.89E-01	3.43E-01	6.16E-01	23.75	2500	91.00	4.40E-00	14.00
	Cr	3000	100.60	9.00E-01	J 2.30E-02	2.28E-02	2.29E-02	0.94	1250	98.80	9.00E-01	234.58
	Co	10000	96.40	8.00E-01	UT 6.31E-01	7.99E-01	6.23E-01	6.72	2500	97.60	8.00E-01	1.03
	Cu	3000	93.80	4.00E-01	UT 4.00E-01	4.00E-01	4.00E-01	0.00	1250	96.90	4.00E-01	1.00
	Fe	3000	97.00	1.00E-00	J 1.31E-02	1.30E-02	1.40E-02	13.37	1250	94.70	1.00E-00	140.47
	Pb	3000	94.00	6.20E-00	UT 6.93E-00	6.93E-00	7.03E-00	23.10	1250	93.20	6.20E-00	1.28
	Mg	3000	93.70	6.60E-01	J 4.23E-00	3.66E-00	3.93E-00	14.41	1250	99.70	3.00E-01	13.16
	Mn	3000	93.60	2.00E-01	J 2.70E-01	2.31E-01	2.31E-01	13.27	1250	93.70	2.00E-01	123.28
	Ni	3000	97.30	1.70E-00	UT 1.70E-00	1.70E-00	1.70E-00	0.00	1250	98.10	1.70E-00	1.00
	K	3000	103.18	1.12E-01	J 7.83E-02	7.83E-02	7.83E-02	0.03	1250	83.10	1.12E-01	69.93
	Se	3000	93.90	7.60E-00	UT 6.44E-00	7.39E-00	6.01E-00	10.33	1250	101.80	7.60E-00	1.03
	Ag	3000	101.00	3.00E-01	UT 1.07E-00	1.26E-00	1.16E-00	16.47	1250	102.50	3.00E-01	2.32
	Na	10000	96.60	2.88E-01	J 3.30E-04	3.31E-04	3.30E-04	0.14	2500	-27.70	3.10E-00	11301.4
	V	10000	93.30	3.00E-01	UT 8.20E-01	3.94E-01	7.07E-01	31.89	2500	97.60	3.00E-01	1.44
	Zn	10000	97.90	3.49E-00	UT 3.00E-01	3.00E-01	3.00E-01	0.00	2500	97.00	3.00E-01	1.00
	Bi	3000	99.60	7.30E-00	J 2.03E-02	1.76E-02	1.91E-02	13.42	1250	39.90	7.30E-00	23.42
	B	3000	87.80	6.00E-01	J 3.12E-00	3.27E-00	3.19E-00	4.72	1250	86.10	6.00E-01	3.32
	Cd	3000	99.00	1.01E-01	UT 1.01E-01	1.01E-01	1.01E-01	0.00	1250	99.30	1.01E-01	1.00
	La	3000	99.10	1.40E-00	J 9.35E-00	7.69E-00	8.52E-00	10.33	1250	100.50	1.40E-00	6.09
	P	10000	93.40	3.80E-00	J 6.11E-03	3.81E-03	3.96E-03	3.01	2500	82.80	3.80E-00	1027.04
	Si	10000	80.00	1.30E-00	J 3.89E-03	3.30E-03	3.60E-03	10.68	2500	99.10	1.30E-00	430.39
	Si	10000	97.80	3.00E-01	J 2.29E-00	1.97E-00	2.13E-00	14.98	2500	99.00	3.00E-01	7.10
	S	3000	97.10	2.70E-00	J 1.21E-03	1.19E-03	1.20E-03	0.96	1250	93.70	2.70E-00	444.44
	Sn	3000	92.70	1.60E-00	UT 1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.60	1.60E-00	1.00
	Ti	3000	97.80	4.00E-01	UT 4.00E-01	4.00E-01	4.00E-01	0.00	1250	99.40	4.00E-01	1.00
	Zr	10000	91.30	8.00E-01	UT 7.99E-01	7.99E-01	7.99E-01	0.00	--	--	--	--

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T-111 Core 31 Composite 2

ID	Analyte	LMCS Std		PREP BLK	Result #1		Result #2	Average	RPD	Spk add		DET LIM	Ratio	Recg	Min
		Added	S Rec		(ug/g)	(ug/g)				S Rec	S Rec		R/DL	Loss	G
11467															
ICP-Fusion	Al	5000	93.30	1.20E-01	J 7.00E-02	6.80E-02	6.93E-02	3.87		1250	94.20	1.20E-01	57.76		
	Si	5000	90.70	8.83E-01	UT 8.83E-01	8.83E-01	8.83E-01	0.00		1250	101.80	8.83E-01	1.00		
	As	5000	91.30	1.50E-01	UT 1.50E-01	1.50E-01	1.50E-01	0.00		2500	44.80	1.50E-01	1.00		
	Ba	10000	97.00	1.50E-00	J 6.13E-01	5.96E-01	6.04E-01	3.09		2500	98.00	1.50E-00	40.37		
	Ba	10000	99.80	1.13E-00	UT 4.99E-01	4.99E-01	4.99E-01	0.00		2500	99.70	1.00E-01	1.00		
	Ca	10000	99.60	2.00E-00	J 1.41E-01	7.18E-00	1.07E-01	61.11		2500	100.40	2.00E-00	3.33		
	Ca	10000	92.40	1.28E-01	J 2.83E-03	2.49E-03	2.66E-03	12.33		2500	94.80	2.20E-01	120.98		
	Cr	5000	102.30	4.50E-00	J 1.73E-03	1.67E-03	1.70E-03	3.37		1250	106.00	4.50E-00	377.27		
	Co	10000	99.10	4.00E-00	UT 1.03E-01	1.11E-01	1.08E-01	4.91		2500	99.60	4.00E-00	2.70		
	Cu	5000	93.60	3.33E-00	J 3.42E-01	3.41E-01	3.42E-01	0.32		1250	96.30	2.00E-00	17.09		
	Fe	5000	97.80	7.13E-01	J 1.97E-04	1.93E-04	1.96E-04	1.36		1250	123.30	3.00E-00	3918.94		
	Pb	5000	91.70	3.10E-01	J 4.84E-03	4.87E-03	4.84E-03	0.89		1250	88.90	3.10E-01	15.82		
	Mg	5000	99.50	1.4E-01	J 4.34E-02	4.31E-02	4.43E-02	3.31		1250	100.90	1.50E-00	295.61		
	Mn	5000	94.60	5.04E-00	J 6.02E-03	5.86E-03	5.94E-03	3.71		1250	103.20	1.00E-00	3941.37		
	Ni	5000	98.80	3.91E-01	J 3.40E-03	7.49E-03	5.43E-03	73.09		1250	107.00	8.50E-00	641.00		
	K	5000	140.10	3.60E-01	UT 3.39E-01	3.39E-01	3.39E-01	0.60		1250	1344.3	3.60E-01	1.00		
	Se	5000	93.20	3.80E-01	UT 3.79E-01	3.79E-01	3.79E-01	0.00		1250	96.60	3.80E-01	1.00		
	Ag	5000	100.80	8.50E-00	J 2.26E-02	2.17E-02	2.21E-02	4.24		1250	103.10	2.50E-00	81.33		
	Na	10000	93.00	4.69E-01	J 3.94E-04	3.83E-04	3.88E-04	2.44		2500	109.80	1.33E-01	2313.61		
	V	10000	98.20	2.50E-00	J 1.74E-01	1.53E-01	1.63E-01	11.33		2500	99.90	2.50E-00	6.60		
	Zn	10000	99.60	1.34E-01	J 1.00E-02	1.11E-02	1.06E-02	9.63		2500	99.80	1.50E-00	70.39		
	Bi	5000	94.40	3.73E-01	J 2.01E-04	2.02E-04	2.01E-04	0.33		1250	79.40	3.73E-01	337.33		
	B	5000	63.10	3.00E-00	UT 2.99E-00	2.99E-00	2.99E-00	0.00		1250	83.10	3.00E-00	1.00		
	Co	5000	101.30	3.03E-01	UT 3.04E-01	3.04E-01	3.04E-01	0.00		1250	97.80	3.03E-01	1.00		
	La	5000	99.50	7.00E-00	J 3.43E-03	3.38E-03	3.41E-03	2.16		1250	103.40	7.00E-00	487.63		
	P	10000	101.90	2.90E-01	J 1.11E-04	1.11E-04	1.11E-04	0.36		2500	103.70	2.90E-01	383.37		
	Si	10000	75.10	6.79E-01	J 3.89E-03	3.78E-03	3.84E-03	1.87		2500	79.10	6.50E-00	897.91	0.43	
	Si	10000	97.30	1.50E-00	J 2.91E-02	2.70E-02	2.80E-02	7.73		2500	98.40	1.50E-00	186.98		
	S	5000	108.00	2.77E-01	J 1.33E-03	1.29E-03	1.31E-03	3.36		1250	102.40	1.33E-01	96.93		
	Sn	5000	92.00	1.16E-00	UT 7.91E-00	7.91E-00	7.91E-00	0.00		1250	93.80	8.00E-00	1.00		
	Ti	5000	96.80	2.00E-00	J 7.34E-03	7.13E-03	7.24E-03	2.88		1250	97.80	2.00E-00	36.18		
	Zr	10000	93.20	4.00E-00	UT 3.99E-00	3.99E-00	3.99E-00	0.00		1250	--	--	--		

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T-111 Core 31 Composite 2

Charge Bal Tot Cal 3.76
Tot Am 2.70
Cu/Am 1.39

Material Balance: oxide model 0.93

Water ICP/Acid ICP

Ca 0.12
Mn 0.00
Si 2.00
S 0.93
Na 0.91
Al 0.01
K 0.61
P 0.60

Water ICP/Fusion ICP

Al 0.015
Cr 0.133
Fe 0.007
Mn 0.004
Ni 0.009
P 0.536
Si 0.096
Sr 0.006

Fusion ICP/Acid ICP

Al 0.96
Fe 0.96
Na 1.01
Ni 0.87
Si 12.39
Ti 2.10
U 1.12
Sr 1.00

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD	Sgt Rec	DET LDM	Ratio	mcg	Mat Bal
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S	S	(ug/g)	R/DL	mcg	(g/g)
Fluor	18 467	104.6	< 1.1E-2	J 3.75E-03	4.00E-03	3.88E-03	0.43	104.4	2.30E-02	13.30	0.033	0.004
	U as uCu/g					1.29E-03						
	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD	Sgt Rec	DET LDM	Ratio	Rel.	
		S Rec	(uCi/g)	(uCi/g)	(uCi/g)	(uCi/g)	S	S	(uCi/g)	R/DL	Ci Err	
A-51	18 467											
R&D-Fus.	TA	101.0	< 1.4E-3	J 3.69E-01	3.30E-01	3.59E-01	3.29	101.3	7.10E-03	30.63	3.10	
	TA/Pu Am					2.01E-00						
	18 467											
	TB	76.10	< 9.1E-2	2.13E-01	2.16E-01	2.15E-01	1.40	76.20	0.35E-02	229.41	1.20	
	TB/Cs, Sr					1.00E-00						
	18 467											
GEA	Ca-133	101.0	7.80E-04	2.38E-01	2.36E-01	2.37E-01	0.84	NA	3.70E-04	640.34	1.100	
	Eu-154	NA	< 9.7E-4	3.24E-03	< 1.06E-3	NA	NA	NA	1.20E-03	NA	33.000	
	Eu-155	NA	< 6.13E-4	< 1.12E-3	< 1.13E-3	NA	NA	NA	3.91E-04	NA	NA	
	Am-241	NA	< 1.17E-3	4.16E-02	4.02E-02	4.09E-02	3.42	NA	0.14E-03	35.81	13.900	
	Co-60	99.60	< 3.6E-4	< 7.75E-4	< 8.5E-4	NA	NA	NA	4.07E-03	NA	NA	
	18 467											
	PU239/40	91.60	< 6.0E-3	J 1.37E-01	1.34E-01	1.34E-01	2.21	91.70	3.60E-03	37.64	0.80	
	PU-238	NA	< 6.0E-3	< 1.04E-3	< 1.03E-3	NA	NA	69.90		NA		

T-111 Core 31 Composite 2

Analyte	Std #1	Prep Blk	Result #1	Result #2	Average	RPD	Std Dev	DET LDI	Ratio	Ref.	
	S Rec	(uCi/g)	(uCi/g)	(uCi/g)	(uCi/g)	%	%	(uCi/g)	R/DL	Q Err	
18-467											
Am-241	100.1	<3.70E-3	J 3.66E-02	4.66E-02	4.31E-02	16.24	9.60	3.70E-03	7.36	10.60	
							11.90			9.60	
Am-241			none detected		NA	NA	NA	NA	NA		
	Am-241 (DETA)/Am-241 (ALPHA)				9.69E-01						
18-467											
NP-237	70.30	<6.49E-3	WS <3.24E-2	<3.24E-2	NA	NA	73.70	3.24E-02	NA	3.20	
18-467											
TC-99	118.3	<8.46E-4	4.36E-03	4.90E-03	4.73E-03	7.10	73.10	8.50E-04	3.36	4.60	
							73.30				
18-467											
I-129	118.3	<1.60E-2	WS <1.92E-2	<2.40E-2	NA	NA	30.20	1.73E-02	NA		
							32.10				

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[illegible]

T-111 Core Composite Summary

Analyte	C31CC1			C31CC2			C31CC1			C31CC2		
	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average
ICP-acid	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Al	3.81E-02	3.80E-02	3.84E-02	7.07E-02	7.03E-02	7.05E-02	4.73E-02	4.71E-02	4.72E-02	4.04E-02	4.03E-02	4.03E-02
Sb	2.19E-01	2.00E-01	2.04E-01	1.69E-01	3.61E-01	3.65E-01	3.18E-01	2.00E-01	3.39E-01	2.61E-01	1.01E-01	3.26E-01
As	1.17E-00	1.13E-00	1.15E-00	1.61E-00	3.07E-00	2.31E-00	3.50E-00	3.39E-00	3.45E-00	2.90E-00	3.23E-00	3.06E-00
Ba	1.73E-01	1.66E-01	1.70E-01	6.46E-01	6.33E-01	6.49E-01	6.70E-01	6.67E-01	6.68E-01	8.79E-01	8.67E-01	8.73E-01
Ba	1.04E-01	1.04E-01	1.04E-01	9.38E-02	1.01E-01	9.73E-02	1.20E-01	1.13E-01	1.17E-01	1.00E-01	1.04E-01	1.04E-01
Ca	7.23E-00	7.19E-00	7.22E-00	7.94E-00	7.78E-00	7.86E-00	4.70E-00	4.09E-00	4.40E-00	3.80E-00	3.64E-00	3.72E-00
Ca	2.36E-03	2.13E-03	2.20E-03	2.61E-03	2.36E-03	2.48E-03	1.30E-03	1.49E-03	1.49E-03	1.34E-03	1.33E-03	1.33E-03
Cr	1.89E-03	1.83E-03	1.86E-03	1.84E-03	1.84E-03	1.84E-03	2.08E-03	2.03E-03	2.04E-03	2.13E-03	2.14E-03	2.14E-03
Co	3.38E-00	3.42E-00	3.40E-00	1.17E-01	3.79E-00	7.76E-00	3.10E-00	3.15E-00	3.13E-00	2.70E-00	3.13E-00	2.91E-00
Cu	2.57E-01	2.44E-01	2.52E-01	3.17E-01	1.77E-02	7.94E-01	1.63E-01	1.64E-01	1.64E-01	1.30E-01	1.20E-01	1.30E-01
Fe	1.95E-04	1.89E-04	1.92E-04	2.00E-04	2.01E-04	2.00E-04	1.76E-04	1.74E-04	1.75E-04	1.72E-04	1.73E-04	1.73E-04
Pb	4.81E-02	4.69E-02	4.75E-02	3.44E-02	3.42E-02	3.43E-02	2.81E-02	2.00E-02	2.81E-02	1.69E-02	1.67E-02	1.68E-02
Mg	4.38E-02	4.32E-02	4.35E-02	4.82E-02	4.73E-02	4.79E-02	3.07E-02	3.02E-02	3.05E-02	2.92E-02	2.88E-02	2.90E-02
Mn	6.31E-03	6.07E-03	6.19E-03	6.14E-03	6.14E-03	6.14E-03	6.77E-03	6.63E-03	6.71E-03	6.23E-03	6.32E-03	6.28E-03
Ni	1.54E-02	1.49E-02	1.51E-02	1.37E-02	1.37E-02	1.37E-02	1.10E-02	1.09E-02	1.10E-02	1.01E-02	1.09E-02	1.01E-02
K	1.11E-03	1.01E-03	1.10E-03	1.20E-03	1.22E-03	1.21E-03	1.22E-03	1.21E-03	1.21E-03	1.02E-03	1.02E-03	1.02E-03
Se	8.04E-00	7.92E-00	7.98E-00	7.13E-00	7.64E-00	7.40E-00	1.20E-01	8.60E-00	1.03E-01	7.40E-00	8.11E-00	7.79E-00
Ag	2.03E-02	2.02E-02	2.03E-02	2.21E-02	2.25E-02	2.27E-02	4.39E-01	4.48E-01	4.43E-01	3.19E-01	3.81E-01	3.00E-01
Na	3.80E-04	3.71E-04	3.76E-04	3.84E-04	3.87E-04	3.87E-04	3.30E-04	3.49E-04	3.50E-04	3.67E-04	3.63E-04	3.63E-04
V	1.31E-03	1.23E-03	1.27E-03	2.13E-03	2.13E-03	2.14E-03	1.42E-03	1.33E-03	1.39E-03	1.04E-03	9.38E-00	9.99E-00
Zn	8.20E-01	7.61E-01	7.94E-01	1.04E-02	9.68E-01	1.01E-02	4.47E-01	4.36E-01	4.42E-01	3.54E-01	3.43E-01	3.50E-01
Bi	2.37E-04	2.34E-04	2.36E-04	2.32E-04	2.33E-04	2.33E-04	2.84E-04	2.84E-04	2.83E-04	2.82E-04	2.84E-04	2.84E-04
B	3.01E-01	3.34E-01	2.71E-01	2.16E-01	2.33E-01	2.34E-01	2.94E-01	2.82E-01	2.94E-01	3.23E-01	3.20E-01	3.22E-01
Co	3.13E-01	3.19E-01	3.26E-01	2.83E-01	2.90E-01	2.86E-01	3.37E-01	4.00E-01	3.78E-01	3.89E-01	3.28E-01	3.31E-01
La	3.78E-03	3.64E-03	3.72E-03	3.19E-03	3.64E-03	3.62E-03	4.67E-03	4.61E-03	4.64E-03	4.86E-03	4.91E-03	4.89E-03
P	1.00E-04	1.02E-04	1.01E-04	9.18E-03	9.94E-03	9.94E-03	9.73E-03	9.87E-03	9.84E-03	1.14E-04	1.13E-04	1.13E-04
Si	4.36E-03	3.29E-02	4.82E-02	3.21E-02	4.18E-02	4.71E-02	4.80E-03	3.73E-02	3.28E-02	2.98E-02	4.90E-02	3.84E-02
Si	2.83E-02	2.78E-02	2.87E-02	2.80E-02	2.80E-02	2.80E-02	3.01E-02	3.01E-02	3.03E-02	3.32E-02	3.37E-02	3.34E-02
S	1.21E-03	1.21E-03	1.21E-03	1.27E-03	1.26E-03	1.26E-03	1.14E-03	1.14E-03	1.14E-03	1.22E-03	1.22E-03	1.22E-03
Se	4.13E-00	4.28E-00	4.21E-00	3.13E-00	3.74E-00	3.44E-00	1.80E-00	1.81E-00	1.81E-00	1.50E-00	1.72E-00	1.61E-00
Ti	2.93E-01	2.94E-01	2.94E-01	3.77E-01	3.34E-01	3.30E-01	8.80E-00	9.00E-00	8.90E-00	4.50E-00	6.42E-00	4.44E-00
Zr	8.47E-01	8.34E-01	8.40E-01	7.30E-01	8.04E-01	7.78E-01	9.20E-01	9.03E-01	9.11E-01	7.70E-01	8.61E-01	8.16E-01

T-111 Core Composite Summary

Analyte	C31CC1			C31CC2			C33CC1			C33CC2		
	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)
DSC	EXOTHER	EXOTHER	NA	EXOTHER	EXOTHER	NA	NO EXO	NO EXO	NA	NO EXO	NO EXO	NA
	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)
TGA	65.30	61.30	73.30	7.12E-01	6.91E-01	7.02E-01	8.21E-01	8.10E-01	8.16E-01	8.30E-01	7.64E-01	8.04E-01
	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)
FWTR	74.60	74.60	74.60	7.59E-01	7.59E-01	7.59E-01	7.51E-01	7.71E-01	7.61E-01	7.64E-01	7.78E-01	7.71E-01
	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)
H ₂	1.48E-00	1.70E-00	1.59	1.88E-00	1.79E-00	1.83E-00	1.72E-00	1.10E-00	1.20E-00	1.15E-00	1.02E-00	1.08E-00
	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)	(N)
CH	<4.0	<4.3	<4.3	<4.37	<4.41	NA	<4.81	<4.90	NA	<4.61	<4.76	NA

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T-111 Core Composite Summary

Analyte	C31CC1			C31CC2			C33CC1			C33CC2		
	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)
ICP - Water												
Al	6.41E-00	6.99E-00	6.71E-00	1.02E-01	1.04E-01	1.03E-01	1.77E-01	1.34E-01	1.55E-01	1.02E-01	1.10E-01	1.10E-01
Sb	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01	1.77E-01
As	3.00E-00	3.00E-00	3.00E-00	3.00E-00	3.00E-00	3.00E-00	2.99E-00	2.99E-00	2.99E-00	3.00E-00	3.00E-00	3.00E-00
Ba	3.00E-01	3.00E-01	3.00E-01	3.32E-01	3.00E-01	4.16E-01	7.15E-01	3.70E-01	5.47E-01	3.01E-01	3.32E-01	3.16E-01
Ba	9.99E-02	1.00E-01	1.00E-01	9.99E-02	9.99E-02	9.99E-02	9.99E-02	9.99E-02	9.99E-02	9.99E-02	9.99E-02	9.99E-02
Ca	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	3.99E-01	3.99E-01	3.99E-01	4.00E-01	3.99E-01	3.99E-01
Ca	5.12E-01	5.05E-01	5.08E-01	6.89E-01	5.45E-01	6.16E-01	6.15E-01	7.16E-01	6.64E-01	4.15E-01	9.35E-01	6.75E-01
Cr	2.07E-02	2.11E-02	2.09E-02	2.30E-02	2.28E-02	2.29E-02	2.26E-02	2.22E-02	2.24E-02	2.09E-02	2.12E-02	2.11E-02
Co	7.99E-01	8.00E-01	8.00E-01	8.31E-01	7.99E-01	8.25E-01	8.45E-01	7.96E-01	8.21E-01	7.99E-01	8.30E-01	8.21E-01
Cu	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	3.99E-01	3.99E-01	3.99E-01	4.00E-01	3.99E-01	3.99E-01
Fe	1.02E-02	1.70E-01	7.94E-01	1.51E-02	1.30E-02	1.40E-02	1.47E-02	1.16E-02	1.32E-02	1.58E-02	1.60E-02	1.59E-02
Pb	6.19E-00	7.91E-00	7.05E-00	6.92E-00	6.93E-00	7.93E-00	6.26E-00	6.19E-00	6.24E-00	6.19E-00	6.19E-00	6.19E-00
Mg	3.22E-00	2.67E-00	2.94E-00	4.23E-00	3.66E-00	3.95E-00	4.06E-00	3.60E-00	3.84E-00	3.72E-00	3.95E-00	3.83E-00
Mn	1.95E-01	1.01E-01	1.47E-01	2.70E-01	2.51E-01	2.51E-01	3.04E-01	3.04E-01	2.54E-01	3.42E-01	3.34E-01	3.38E-01
Ni	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00	1.70E-00
K	7.24E-02	7.40E-02	7.34E-02	7.83E-02	7.83E-02	7.83E-02	7.19E-02	7.04E-02	7.12E-02	6.30E-02	6.47E-02	6.48E-02
Se	7.59E-00	7.60E-00	7.60E-00	8.44E-00	7.59E-00	8.01E-00	7.58E-00	7.56E-00	7.58E-00	7.59E-00	7.58E-00	7.59E-00
Ag	4.99E-01	7.79E-01	6.39E-01	1.07E-00	1.26E-00	1.16E-00	6.08E-01	4.99E-01	5.53E-01	4.99E-01	4.99E-01	4.99E-01
Na	3.39E-04	3.41E-04	3.40E-04	3.30E-04	3.31E-04	3.30E-04	3.09E-04	3.06E-04	3.08E-04	3.22E-04	3.19E-04	3.20E-04
V	4.99E-01	3.00E-01	3.00E-01	8.20E-01	5.94E-01	7.07E-01	4.99E-01	4.99E-01	4.99E-01	6.76E-01	9.21E-01	7.99E-01
Zn	3.00E-01	3.00E-01	3.00E-01	3.00E-01	3.00E-01	3.00E-01	2.99E-01	2.99E-01	2.99E-01	3.00E-01	2.99E-01	3.00E-01
Bi	1.47E-02	8.34E-01	1.15E-02	2.05E-02	1.76E-02	1.91E-02	2.58E-02	2.05E-02	2.31E-02	2.67E-02	2.75E-02	2.70E-02
B	3.11E-00	3.30E-00	3.31E-00	3.12E-00	3.27E-00	3.19E-00	3.54E-00	3.54E-00	3.54E-00	4.06E-00	4.44E-00	4.25E-00
Co	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01	1.01E-01
La	7.05E-00	3.00E-00	6.02E-00	9.35E-00	7.69E-00	8.52E-00	8.55E-01	8.22E-01	8.38E-01	1.58E-01	1.97E-01	1.58E-01
P	3.99E-03	3.83E-03	3.76E-03	6.11E-03	5.81E-03	5.96E-03	5.34E-03	5.26E-03	5.30E-03	5.74E-03	5.66E-03	5.70E-03
M	3.30E-02	3.45E-02	4.38E-02	3.89E-02	3.30E-02	3.60E-02	6.71E-02	6.68E-02	6.69E-02	6.18E-02	6.22E-02	6.20E-02
Sr	1.46E-00	9.34E-01	1.21E-00	2.29E-00	1.97E-00	2.13E-00	2.36E-00	1.97E-00	2.18E-00	2.29E-00	2.39E-00	2.34E-00
S	1.18E-03	1.21E-03	1.19E-03	1.21E-03	1.19E-03	1.20E-03	1.07E-03	1.05E-03	1.06E-03	1.14E-03	1.14E-03	1.14E-03
Se	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00	1.60E-00
Ti	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	4.00E-01	3.99E-01	3.99E-01	3.99E-01	4.00E-01	3.99E-01	3.99E-01
Zr	7.99E-01	8.00E-01	8.00E-01	7.99E-01	7.99E-01	7.99E-01	7.98E-01	7.98E-01	7.98E-01	7.99E-01	7.98E-01	7.99E-01

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T-111 Core Composite Summary

T-III Core Composite Summary

Analyte	C33CC1			C33CC2			C33CC1			C33CC2		
	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average
ICP Fusion	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Al	6.56E-02	6.32E-02	6.44E-02	7.04E-02	6.80E-02	6.92E-02	4.83E-02	4.83E-02	4.84E-02	4.39E-02	4.39E-02	4.39E-02
Sb	8.83E-01	8.87E-01	8.84E-01	6.83E-01	8.83E-01	8.83E-01	8.83E-01	1.29E-02	1.09E-02	8.83E-01	8.83E-01	8.84E-01
As	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01	1.30E-01
Ba	6.04E-01	5.72E-01	5.88E-01	6.13E-01	5.94E-01	6.04E-01	6.48E-01	6.40E-01	6.44E-01	7.30E-01	7.41E-01	7.37E-01
Ba	5.00E-01	5.01E-01	5.01E-01	4.99E-01	4.99E-01	4.99E-01	5.00E-01	4.99E-01	4.99E-01	4.99E-01	5.00E-01	4.99E-01
Ca	9.16E-00	7.34E-00	8.23E-00	1.41E-01	7.18E-00	1.07E-01	6.08E-00	6.74E-00	6.42E-00	7.44E-00	6.84E-00	7.17E-00
Ca	2.91E-03	2.36E-03	2.74E-03	2.83E-03	2.49E-03	2.64E-03	1.93E-03	2.30E-03	2.22E-03	1.92E-03	2.18E-03	2.03E-03
Cr	1.92E-03	1.84E-03	1.89E-03	1.73E-03	1.47E-03	1.70E-03	1.74E-03	1.81E-03	1.79E-03	1.82E-03	1.82E-03	1.82E-03
Ca	1.03E-01	9.70E-00	1.01E-01	1.03E-01	1.11E-01	1.04E-01	1.30E-01	1.37E-01	1.33E-01	1.48E-01	8.80E-00	1.18E-01
Cu	3.39E-01	3.88E-01	3.63E-01	3.42E-01	3.41E-01	3.42E-01	2.24E-01	2.14E-01	2.21E-01	2.41E-01	2.30E-01	2.44E-01
Fe	2.08E-04	2.02E-04	2.03E-04	1.97E-04	1.93E-04	1.94E-04	1.37E-04	1.62E-04	1.39E-04	1.61E-04	1.61E-04	1.61E-04
Pb	4.33E-02	4.37E-02	4.40E-02	4.84E-02	4.82E-02	4.84E-02	2.72E-02	2.62E-02	2.67E-02	2.67E-02	2.72E-02	2.69E-02
Mg	4.32E-02	4.34E-02	4.38E-02	4.34E-02	4.31E-02	4.43E-02	2.18E-02	2.78E-02	2.48E-02	2.49E-02	2.74E-02	2.72E-02
Mn	6.47E-03	6.29E-03	6.38E-03	6.02E-03	5.84E-03	5.94E-03	6.13E-03	6.29E-03	6.22E-03	6.39E-03	6.39E-03	6.39E-03
Ni	3.61E-03	3.14E-03	3.77E-03	3.40E-03	7.49E-03	3.43E-03	9.09E-03	9.49E-03	9.29E-03	1.43E-04	9.81E-03	1.21E-04
K	3.60E-01	3.61E-01	3.61E-01	3.39E-01	3.39E-01	3.39E-01	3.60E-01	3.39E-01	3.39E-01	3.39E-01	3.60E-01	3.39E-01
Se	3.80E-01	3.81E-01	3.80E-01	3.79E-01	3.79E-01	3.79E-01	3.80E-01	3.79E-01	3.80E-01	3.79E-01	3.80E-01	3.80E-01
Ag	2.18E-02	2.10E-02	2.14E-02	2.36E-02	2.17E-02	2.21E-02	4.00E-01	3.89E-01	3.93E-01	3.74E-01	3.69E-01	3.71E-01
Na	4.01E-04	3.94E-04	3.98E-04	3.94E-04	3.83E-04	3.90E-04	3.34E-04	3.41E-04	3.39E-04	3.32E-04	3.31E-04	3.32E-04
V	1.32E-01	1.11E-01	1.21E-01	1.74E-01	1.33E-01	1.63E-01	1.69E-01	1.38E-01	1.33E-01	1.31E-01	1.42E-01	1.47E-01
Zn	1.11E-02	9.71E-01	1.04E-02	1.00E-02	1.11E-02	1.04E-02	1.00E-02	1.10E-02	1.03E-02	1.10E-02	1.07E-02	1.10E-02
Bi	2.14E-04	2.03E-04	2.09E-04	2.01E-04	2.02E-04	2.01E-04	2.63E-04	2.64E-04	2.63E-04	2.61E-04	2.73E-04	2.67E-04
B	3.00E-00	3.01E-00	3.00E-00	2.99E-00	2.99E-00	2.99E-00	4.32E-00	3.34E-00	4.84E-00	3.10E-00	4.38E-00	4.84E-00
Co	3.03E-01	3.04E-01	3.04E-01	3.04E-01	3.04E-01	3.04E-01	3.03E-01	3.04E-01	3.04E-01	3.04E-01	3.03E-01	3.04E-01
La	3.73E-03	3.63E-03	3.69E-03	3.43E-03	3.38E-03	3.41E-03	4.43E-03	4.36E-03	4.31E-03	4.78E-03	4.84E-03	4.81E-03
P	1.19E-04	1.12E-04	1.14E-04	1.21E-04	1.11E-04	1.11E-04	9.13E-03	8.99E-03	9.07E-03	9.01E-03	9.01E-03	9.01E-03
M	6.04E-03	5.88E-03	5.94E-03	5.89E-03	5.78E-03	5.84E-03	3.39E-03	3.32E-03	3.44E-03	3.41E-03	3.41E-03	3.41E-03
Se	3.98E-02	3.08E-02	3.03E-02	3.01E-02	2.70E-02	2.80E-02	2.84E-02	2.97E-02	2.91E-02	3.34E-02	2.98E-02	3.17E-02
S	1.34E-03	1.38E-03	1.33E-03	1.33E-03	1.29E-03	1.31E-03	1.01E-03	1.04E-03	1.04E-03	1.14E-03	1.14E-03	1.14E-03
Se	8.00E-00	8.02E-00	8.01E-00	7.98E-00	7.98E-00	7.98E-00	8.00E-00	7.98E-00	7.99E-00	7.98E-00	8.00E-00	7.99E-00
Ti	7.23E-01	7.32E-01	7.29E-01	7.34E-01	7.13E-01	7.24E-01	2.22E-01	2.23E-01	2.23E-01	2.31E-01	2.31E-01	2.41E-01
Zr	4.00E-00	4.01E-00	4.00E-00	3.99E-00	3.99E-00	3.99E-00	4.00E-00	3.99E-00	4.00E-00	3.99E-00	4.00E-00	4.00E-00

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T-III Core Composite Summary

Analysis	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average	Result #1	Result #2	Average
50-90	7.14E-00	6.97E-00	7.16E-00	(PCV)	(PCV)		7.11E-00	7.11E-00	7.11E-00	7.11E-00	7.11E-00	7.11E-00	7.11E-00	7.11E-00	7.11E-00
H-1	Q.11E-4	Q.11E-4	NA	Q.11E-4	Q.11E-4	NA	Q.11E-4	Q.11E-4	NA	Q.11E-4	Q.11E-4	NA	Q.11E-4	Q.11E-4	NA
C-14	Q.21E-4	2.80E-04	NA	Q.21E-4	Q.21E-4	NA	Q.21E-4	Q.21E-4	NA	Q.21E-4	Q.21E-4	NA	Q.21E-4	Q.21E-4	NA
54-78	<4.11E-3	<4.05E-3	NA	<4.11E-3	<4.11E-3	NA	<4.11E-3	<4.11E-3	NA	<4.11E-3	<4.11E-3	NA	<4.11E-3	<4.11E-3	NA

T-111 Core 33 Direct Segment Analyses

Segment 1

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10.310						
DSC	EXOTHERM	NA	EXOTHERM	EXOTHERM	NA	NA
10.310						
TOA	99.3	NA	77.8	NA	NA	NA
10.310						
S WATER	100.7	NA	81.1	79.6	80.4	1.87

Segment 2

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10.323						
DSC	EXOTHERM	NA	EXOTHERM	EXOTHERM	NA	NA
10.323						
TOA	99.3	NA	80.3	80.6	80.4	0.12
10.323						
S WATER	98.1	NA	83.6	83.8	83.7	0.23

Segment 3

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10.324						
DSC	EXOTHERM	NA	EXOTHERM	NO EXO	NA	NA
10.324						
TOA	99.3	NA	81.3	NA	NA	NA
10.324						
S WATER	98.1	NA	81.7	82.9	81.9	0.33

T-111 Core 33 Direct Segment Analyses

Segment 4

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10323						
DSC	EXOTHERM	NA	NO EXO	NA	NA	NA
10323						
IGA	99.3	NA	89.3	NA	NA	NA
10323						
WATER	98.1	NA	80.4	79.3	79.9	1.38

Segment 5

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10326						
DSC	EXOTHERM	NA	NO EXO	NA	NA	NA
10326						
IGA	99.3	NA	98.0	NA	NA	NA
10326						
WATER	98.1	NA	79.3	77.0	78.2	2.94

Segment 6

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
10329						
DSC	EXOTHERM	NA	NO EXO	NO EXO	NA	NA
10329						
IGA	94.6	NA	84.7	84.0	84.4	0.83
10329						
WATER	101.0		78.3	78.6	78.5	0.38

T-111 Core 33 Direct Segment Analyses

Segment 7

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
18.330						
DSC	EXOTHERM	NA	NO EXO	NA	NA	NA
18.330						
TGA	94.6	NA	85.8	NA	NA	NA
18.330						
S WATER	101.0	NA	74.7	68.6	71.7	0.31

Segment 8

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
18.331						
DSC	EXOTHERM	NA	NO EXO	NA	NA	NA
18.331						
TGA	94.6	NA	84.8	NA	NA	NA
18.331						
S WATER	101.0	NA	75.4	no sample	NA	NA

Segment 9

Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD
	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S
18.332						
DSC	EXOTHERM	NA	NO EXO	NO EXO	NA	NA
18.332						
TGA	94.6	NA	85.2	NA	NA	NA
18.332						
S WATER	101.0	NA	77.0	74.9	76.0	2.76

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Analyte		1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	16000	17000	18000	19000	20000	21000	22000	23000	24000	25000	26000	27000	28000	29000	30000	31000	32000	33000	34000	35000	36000	37000	38000	39000	40000	41000	42000	43000	44000	45000	46000	47000	48000	49000	50000	51000	52000	53000	54000	55000	56000	57000	58000	59000	60000	61000	62000	63000	64000	65000	66000	67000	68000	69000	70000	71000	72000	73000	74000	75000	76000	77000	78000	79000	80000	81000	82000	83000	84000	85000	86000	87000	88000	89000	90000	91000	92000	93000	94000	95000	96000	97000	98000	99000	100000
1000	1100	1200	1300	1400	1500	1600	1700	1800	1900	2000	2100	2200	2300	2400	2500	2600	2700	2800	2900	3000	3100	3200	3300	3400	3500	3600	3700	3800	3900	4000	4100	4200	4300	4400	4500	4600	4700	4800	4900	5000	5100	5200	5300	5400	5500	5600	5700	5800	5900	6000	6100	6200	6300	6400	6500	6600	6700	6800	6900	7000	7100	7200	7300	7400	7500	7600	7700	7800	7900	8000	8100	8200	8300	8400	8500	8600	8700	8800	8900	9000	9100	9200	9300	9400	9500	9600	9700	9800	9900	10000											

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ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF-A11	11 401	ICF
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WHC-EP-0806
WHC-SD-WM-DP-024 ADDENDUM 2, REV 0

ID 408	Analyte	IACS Std		PREP BLK		Result #1	Result #2	Average	RPD	Spk add	Spike	DET ID4	Ratio
		u.k.k.d	% Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	(ug/g)	% Rec	(ug/g)	R/DL
ICT-Acid	Al UJ	1000	114.4	7.03E-01	1.68E-02	1.55E-02	1.62E-02	7.99	118.76	137.7	2.40E-00	67.60	
	Sh UJ	1000	94.9	1.77E-01	2.61E-01	2.13E-01	2.41E-01	22.92	118.76	91.7	1.77E-01	1.34	
	As UJ	1000	92.3	1.00E-00	4.55E-00	3.61E-00	4.08E-00	22.92	117.76	93.8	1.00E-00	1.34	
	Ba UJ	2000	90.6	3.51E-01	1.95E-01	2.46E-01	2.70E-01	18.04	237.53	88.3	1.00E-01	90.81	
	Be UJ	2000	89.9	1.00E-01	1.51E-01	1.20E-01	1.34E-01	22.78	237.53	84.9	1.00E-01	1.34	
	Ca UJ	2000	91.3	4.00E-01	3.71E-00	3.38E-00	3.54E-00	18.23	237.53	81.4	4.00E-01	8.9	
	Co UJ	2000	138.3	4.09E-01	1.04E-03	1.04E-03	1.04E-03	4.48	237.53	147.3	4.40E-00	240.99	
	Cr UJ	1000	97.9	9.00E-01	2.32E-03	2.43E-03	2.48E-03	2.43	118.76	125.7	9.00E-01	2750.59	
	Cu UJ	2000	92.9	8.00E-01	3.03E-00	2.47E-00	2.76E-00	21.18	237.53	84.4	8.00E-01	3.43	
	Fe UJ	1000	99.7	3.73E-00	1.87E-04	1.76E-04	1.79E-04	3.83	118.76	-245.7	1.00E-00	17947.0	
	Pb UJ	1000	92.8	4.20E-00	1.20E-02	1.18E-02	1.19E-02	2.20	118.76	95.1	6.20E-00	19.20	
	Mg UJ	1000	97.8	3.10E-00	2.04E-02	2.05E-02	2.04E-02	8.33	118.76	107.3	1.00E-01	683.68	
	Mn UJ	1000	89.3	2.00E-01	4.18E-03	4.04E-03	4.12E-03	3.92	118.76	2184.2	2.00E-01	20403.1	
	Ni UJ	1000	91.3	1.70E-00	7.04E-01	6.84E-01	6.98E-01	2.90	118.76	93.8	1.70E-00	41.04	
	K UJ	1000	104.3	1.12E-01	6.81E-02	6.84E-02	6.88E-02	1.03	118.76	137.1	1.12E-01	61.42	
	Se UJ	1000	80.9	7.60E-00	1.13E-01	9.16E-00	1.03E-01	22.82	118.76	81.3	7.60E-00	1.34	
	Ag UJ	1000	101.2	3.00E-01	1.46E-00	1.44E-00	1.55E-00	11.28	118.76	49.3	3.00E-01	3.18	
	Na UJ	2000	127.9	6.41E-01	3.61E-04	3.55E-04	3.58E-04	1.43	237.53	610.8	3.10E-00	11554.9	
	V UJ	2000	94.4	3.00E-01	3.23E-00	4.11E-00	4.68E-00	24.30	237.53	90.7	3.00E-01	9.34	
	Zn UJ	2000	92.7	1.32E-00	2.32E-01	2.30E-01	2.31E-01	8.94	237.53	84.8	1.00E-01	77.00	
	Bi UJ	1000	7.8	-4.82E-01	3.47E-04	3.40E-04	3.43E-04	1.93	118.76	182.8	7.50E-00	4376.23	
	B UJ	1000	73.4	2.23E-01	9.09E-01	7.32E-01	8.16E-01	22.82	118.76	63.8	6.00E-01	1.34	
	C UJ	1000	108.3	1.01E-01	3.84E-01	3.42E-01	3.64E-01	12.13	118.76	109.4	1.01E-01	3.60	
	La UJ	1000	100.4	1.40E-00	4.81E-03	4.81E-03	4.88E-03	2.98	118.76	86.1	1.40E-00	3487.83	
	P UJ	2000	89.6	2.37E-01	1.22E-04	1.20E-04	1.21E-04	1.23	237.53	53.3	3.00E-00	2089.57	
	Si UJ	2000	34.4	1.39E-01	4.23E-02	4.84E-02	4.13E-02	4.31	237.53	134.9	1.30E-00	317.81	
	Sn UJ	2000	90.4	3.00E-01	6.93E-02	4.20E-02	5.56E-02	49.07	237.53	87.8	3.00E-01	1854.04	
	S UJ	1000	113.9	1.49E-01	1.23E-03	1.32E-03	1.22E-03	8.89	118.76	100.9	2.70E-00	452.33	
	Sb UJ	1000	97.6	1.69E-01	2.50E-01	1.95E-01	2.22E-01	24.31	118.76	83.0	1.60E-00	11.90	
	Ti UJ	1000	84.1	5.51E-01	2.53E-00	2.46E-00	2.50E-00	3.47	118.76	89.8	4.00E-01	6.26	
	Zr UJ	2000	90.3	8.00E-01	1.21E-00	9.64E-01	1.09E-00	22.81	237.53	60.9			

1-111 Hong Kong Airlines Analysis For Core 33

[illegible]

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F-111 Home Organization Summary

[illegible]

Table 1: Homogenization Summary for Core 33

Analyte	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	RPD %	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	RPD %	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	RPD %	Result #1 (ug/g)	Result #2 (ug/g)	Average (ug/g)	RPD %
Al	398 239	430 234	414 236	7.723	416 324	411 693	414 109	1.167	416 324	411 693	414 109	1.167	416 324	411 693	414 109	1.167
Si	28 548	27 826	28 189	2.548	19 647	20 392	20 029	3.620	19 647	20 392	20 029	3.620	19 647	20 392	20 029	3.620
As	4 839	4 717	4 778	2.547	4 143	4 143	4 143	0.000	4 143	4 143	4 143	0.000	4 143	4 143	4 143	0.000
Ba	58 060	64 810	61 435	10.987	61 714	60 176	60 945	2.523	61 714	60 176	60 945	2.523	61 714	60 176	60 945	2.523
Bc	0 181	0 137	0 159	2.573	0 111	0 111	0 111	0.000	0 111	0 111	0 111	0.000	0 111	0 111	0 111	0.000
Ca	22 989	15 325	19 156	40.018	14 253	14 122	14 187	0.922	14 253	14 122	14 187	0.922	14 253	14 122	14 187	0.922
Co	4494 063	5193 290	4843 676	14.436	4928 149	4876 215	4902 182	3.083	4928 149	4876 215	4902 182	3.083	4928 149	4876 215	4902 182	3.083
Cr	460 304	512 342	486 324	10.704	502 833	480 163	491 358	4.634	502 833	480 163	491 358	4.634	502 833	480 163	491 358	4.634
Cu	4 830	4 740	4 785	1.073	4 833	4 833	4 833	0.000	4 833	4 833	4 833	0.000	4 833	4 833	4 833	0.000
Fe	88 378	89 894	89 234	1.475	84 235	81 242	82 748	3.710	84 235	81 242	82 748	3.710	84 235	81 242	82 748	3.710
Pb	22189 814	16310 374	18449 743	23.191	15980 423	16202 702	16091 563	1.311	15980 423	16202 702	16091 563	1.311	15980 423	16202 702	16091 563	1.311
Mg	817 307	872 844	845 076	12.740	854 192	835 476	844 834	2.471	854 192	835 476	844 834	2.471	854 192	835 476	844 834	2.471
Mn	758 221	841 337	799 779	10.592	802 351	780 182	791 266	1.378	802 351	780 182	791 266	1.378	802 351	780 182	791 266	1.378
Ni	21318 701	23188 678	22253 689	15.632	24066 667	24608 293	24337 481	2.223	24066 667	24608 293	24337 481	2.223	24066 667	24608 293	24337 481	2.223
K	231 851	248 443	239 167	11.523	233 840	229 462	231 701	3.704	233 840	229 462	231 701	3.704	233 840	229 462	231 701	3.704
Se	1019 127	1123 725	1071 426	9.742	1113 507	1112 899	1113 253	0.404	1113 507	1112 899	1113 253	0.404	1113 507	1112 899	1113 253	0.404
Ag	12 138	11 850	12 104	2.548	8 444	8 756	8 600	3.621	8 444	8 756	8 600	3.621	8 444	8 756	8 600	3.621
Na	472 014	404 344	438 179	4.272	744 321	314 018	544 166	61.846	744 321	314 018	544 166	61.846	744 321	314 018	544 166	61.846
V	21078 897	23973 204	22026 052	8.600	22892 284	22811 301	22851 794	0.354	22892 284	22811 301	22851 794	0.354	22892 284	22811 301	22851 794	0.354
Zn	14 723	16 723	15 723	12.715	17 150	17 060	17 105	0.524	17 150	17 060	17 105	0.524	17 150	17 060	17 105	0.524
Bi	257 850	284 510	271 080	9.909	268 980	265 158	267 069	0.605	268 980	265 158	267 069	0.605	268 980	265 158	267 069	0.605
B	703 854	781 498	742 678	10.709	704 229	744 500	724 364	2.287	704 229	744 500	724 364	2.287	704 229	744 500	724 364	2.287
Cs	0 968	0 943	0 956	2.549	0 647	0 691	0 679	3.609	0 647	0 691	0 679	3.609	0 647	0 691	0 679	3.609
La	16 290	18 421	17 856	17.532	15 322	11 636	13 579	28.619	15 322	11 636	13 579	28.619	15 322	11 636	13 579	28.619
P	2 258	3 204	2 732	34.899	2 974	1 613	2 293	39.335	2 974	1 613	2 293	39.335	2 974	1 613	2 293	39.335
Si	4213 321	4507 321	4360 371	6.731	4229 806	4309 807	4269 807	6.410	4229 806	4309 807	4269 807	6.410	4229 806	4309 807	4269 807	6.410
Sr	436 230	434 400	435 315	0.426	332 346	319 925	326 084	3.778	332 346	319 925	326 084	3.778	332 346	319 925	326 084	3.778
Sr	98 976	109 377	104 277	10.167	103 937	102 138	103 038	1.747	103 937	102 138	103 038	1.747	103 937	102 138	103 038	1.747
Sr	750 638	837 490	794 060	9.741	802 384	801 108	801 844	0.164	802 384	801 108	801 844	0.164	802 384	801 108	801 844	0.164
Se	26 034	31 327	28 681	1.969	21 408	18 519	20 043	15.397	21 408	18 519	20 043	15.397	21 408	18 519	20 043	15.397
Ti	68 993	79 681	74 342	6.378	70 401	68 439	69 420	1.639	70 401	68 439	69 420	1.639	70 401	68 439	69 420	1.639
Zr	1 290	1 250	1 274	2.543	0 889	0 922	0 905	3.623	0 889	0 922	0 905	3.623	0 889	0 922	0 905	3.623
TA	0 623	0 672	0 648	7.568	0 670	0 632	0 651	5.817	0 670	0 632	0 651	5.817	0 670	0 632	0 651	5.817
Co-137	0 406	0 407	0 407	0.246	0 396	0 400	0 398	1.005	0 396	0 400	0 398	1.005	0 396	0 400	0 398	1.005
Eu-154	0 021	0 021	0 021	1.860	0 021	0 021	0 021	1.896	0 021	0 021	0 021	1.896	0 021	0 021	0 021	1.896
Eu-155	0 028	0 027	0 028	0.717	0 027	0 027	0 027	1.449	0 027	0 027	0 027	1.449	0 027	0 027	0 027	1.449
Am-241	0 136	0 136	0 136	0.000	0 140	0 139	0 140	0.717	0 140	0 139	0 140	0.717	0 140	0 139	0 140	0.717
Co-60	0 006	0 006	0 006	7.553	0 005	0 005	0 005	0.513	0 005	0 005	0 005	0.513	0 005	0 005	0 005	0.513

Analyte	Result #1	Result #2	Average	RPD #	Average	RPD #
Al	546 102	544 127	545 115	0.947	0.947	2.914
Sb	16 889	16 454	16 172	2.811	2.811	34 379
As	2 643	2 835	2 739	0.996	0.996	34 322
Ba	25 076	24 833	24 955	0.976	0.976	3 393
Bc	0.095	0.096	0.096	2.481	2.481	34 376
Ca	2 283	2 056	2 169	10.420	10.420	6 419
Ca	1109 020	1164 314	1136 667	1.645	1.645	4 431
Cr	1222 856	1202 617	1212 736	11.648	11.648	7 765
Co	3 485	3 357	3 421	11.744	11.744	3 648
Cu	6 433	6 507	6 470	11.139	11.139	3 281
Pb	1204 677	1189 314	1196 995	1.264	1.264	3 281
Pb	104 377	113 563	108 949	0.430	0.430	4 787
Mg	301 902	301 435	301 619	0.172	0.172	5 724
Mg	3644 776	3817 161	3841 969	1.291	1.291	4 832
Ni	70 038	68 590	69 314	2.117	2.117	1 375
K	1631 684	1631 727	1631 706	0.003	0.003	1 375
Na	7 232	7 434	7 344	2.312	2.312	34 321
Na	26217 178	26177 746	26197 462	2.558	2.558	19 360
V	29 960	29 437	29 708	1.692	1.692	2 979
Zn	15 547	16 570	16 058	6.368	6.368	0 409
Bi	24792 882	24611 682	24702 282	0.754	0.754	4 217
B	0.573	0.587	0.580	2.518	2.518	34 318
Co	37 144	36 838	36 991	0.825	0.825	0 144
La	4025 143	4001 343	4014 217	0.543	0.543	5 383
P	4800 460	4777 229	4788 845	0.483	0.483	1 189
Si	371 109	383 322	377 218	1.238	1.238	23 996
Sc	177 204	175 703	176 453	0.831	0.831	2 362
S	729 309	729 864	729 586	1.272	1.272	2 035
Sn	8 218	8 521	8 370	14.684	14.684	27 162
Ti	4 878	4 391	4 635	170 287	170 287	131 258
Zr	0.763	0.783	0.773	2.509	2.509	34 316
TA	0.197	0.205	0.201	3.980	3.980	42 169
Co-137	0.137	0.137	0.137	0.000	0.000	3 584
Eu-154	0.001	0.001	0.001	1.156	1.156	NA
Eu-155	0.002	0.002	0.002	0.000	0.000	41 975
Am-241	0.014	0.014	0.014	0.712	0.712	1 795
Co-60	0.000	0.000	0.000	NA	NA	200 000

Table Homoculation Summary for Core 33

Analyte	Result #1		Result #2		average		RPD %		Result #1		Result #2		average		RPD %		RPD %	
	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)			(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)			average	average
Al	625 428	645 231	635 329	3 117	607 807	584 070	3 939	3 983	4 398									
Sb	37 065	43 838	40 201	15 603	42 946	30 814	34 479	35 446	0 708									
As	4 021	3 623	3 822	10 418	3 948	4 423	4 187	10 878	9 334									
Ba	181 231	187 930	184 581	3 629	181 337	173 835	177 706	4 334	3 785									
Ba	0 134	0 121	0 127	10 361	0 133	0 148	0 140	10 865	9 353									
Ca	2 892	3 036	2 964	4 862	3 002	3 071	3 037	2 272	2 441									
Ca	1289 490	1329 977	1314 734	3 840	1301 366	1290 219	1295 893	0 876	1 443									
Cr	2030 715	2101 636	2066 175	3 433	2006 901	1947 799	1977 350	2 989	4 393									
Co	13 093	3 341	8 218	118 894	3 499	11 535	13 517	1 029	80 613									
Cu	7 607	8 330	7 969	8 076	8 198	7 516	7 867	8 415	1 282									
Fe	16139 300	16749 333	16434 312	3 384	15972 805	15476 965	15724 885	3 133	4 334									
Pb	106 845	130 184	118 494	19 643	114 906	113 880	114 393	0 897	3 332									
Mg	219 387	234 103	226 746	6 491	227 440	209 943	218 692	8 000	3 616									
Mn	2798 826	2898 282	2848 039	3 741	2764 477	2651 601	2708 014	4 166	4 935									
Ni	85 647	89 347	87 517	4 182	83 983	96 126	90 055	13 484	2 838									
K	1057 387	1093 713	1076 350	3 560	1080 519	1020 431	1050 475	5 720	2 452									
Se	10 188	9 179	9 683	10 420	10 831	11 209	10 631	10 878	0 333									
Ag	1 409	1 123	1 266	22 670	1 316	0 789	1 052	50 076	10 391									
Na	31803 141	32875 630	32340 386	3 310	32919 277	31920 412	31969 844	5 940	1 132									
V	10 293	11 942	11 120	14 871	10 784	12 308	11 546	41 662	33 187									
Zn	19 394	19 979	19 786	1 943	19 474	19 718	19 681	1 398	0 887									
Bi	33330 984	34576 812	34053 898	3 071	33445 763	32185 318	32780 647	4 058	3 810									
B	0 804	0 725	0 764	10 426	0 794	0 885	0 839	10 877	9 340									
Co	44 829	49 481	47 805	7 813	49 237	44 725	46 981	9 603	1 739									
La	5153 255	5355 621	5254 442	3 851	5124 363	4981 343	5017 854	4 245	4 606									
P	8764 883	8900 917	8872 900	2 433	8858 831	9019 846	8990 338	5 581	4 693									
Si	363 410	347 394	356 402	3 055	380 390	333 395	457 093	33 474	24 755									
Sr	321 831	332 599	326 875	3 302	320 291	306 871	313 581	4 280	4 131									
S	1039 230	1065 834	1048 332	3 238	1065 731	1019 630	1042 681	4 421	2 449									
Sn	13 716	13 926	13 821	1 517	13 093	13 566	13 329	3 085	10 346									
Ti	2 004	2 476	2 240	21 075	1 239	3 082	2 171	44 007	3 147									
Zr	1 072	0 966	1 019	10 419	1 034	1 180	1 119	10 875	9 330									
TA	0 531	0 560	0 546	1 620	0 468	0 526	0 496	12 048	10 916									
Cr-137	0 091	0 086	0 089	3 650	0 091	0 086	0 089	3 650	0 000									
Eu-154	<1 E-4	<1 E-4	NA	NA	<1 E-4	<1 E-4	NA	NA	NA									
Eu-153	<1 E-4	<1 E-4	NA	NA	<1 E-4	<1 E-4	NA	NA	NA									
Am-241	0 021	0 020	0 021	4 878	0 021	0 020	0 021	4 878	0 000									
Co-60	<1 E-3	<1 E-4	NA	NA	<1 E-3	<1 E-4	NA	NA	NA									

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THE HONGKONG & SHANGHAI SHIPPING CO. LTD.

[illegible]

Analysis	Result #1	Result #2	Average	RPD #
AI	120 171	119 207	120 189	0 427
SB	31 068	31 325	31 197	10 769
AO	3 896	3 615	3 755	2 073
BA	251 002	251 984	251 493	4 068
CD	0 120	0 121	0 121	2 090
CA	9 209	9 179	9 194	12 408
CC	952 272	946 320	949 296	0 909
CC	2010 815	1993 807	2006 361	0 042
CD	2 607	2 619	2 613	1 693
CO	2 196	2 706	2 451	3 452
FO	10644 551	10518 901	10641 726	0 550
PS	141 074	135 517	138 296	0 130
MF	253 480	222 057	237 769	3 794
FL	4645 892	4642 783	4644 338	0 000
HI	811 684	87 590	10 043	1 920
RI	484 811	475 616	480 228	1 630
SA	0 870	0 157	0 513	0 073
AG	1 243	1 244	1 243	1 630
MA	40655 018	40199 628	40427 323	4 266
V	1 298	1 039	1 169	1 528
ZM	22 728	23 560	23 144	7 110
BI	24303 680	23810 195	24006 938	0 704
B	0 779	0 723	0 751	2 072
CA	43 143	43 616	43 379	3 328
LI	4225 071	4243 268	4234 110	2 352
P	13500 325	14432 392	13964 459	1 079
SI	507 870	448 946	518 408	20 498
SV	329 592	375 206	377 320	0 492
8	1458 906	1416 953	1436 430	1 399
SN	21 415	18 813	20 119	4 963
TI	3 270	2 996	3 133	3 605
ZF	1 039	0 964	1 001	2 078
TA	0 273	0 235	0 253	5 158
CA-127	0 014	0 013	0 013	0 371
EU-154	< 0.02E-4	0 001	NA	NA
EU-191	< 0.02E-4	NA	NA	NA
AM-241	0 051	0 050	0 050	0 197
Co-60	< 0.02E-3	NA	NA	NA
Result #1	Result #2	Average	RPD #	
AI	126 449	126 442	126 445	0 599
SB	21 290	21 114	21 202	10 634
AO	3 409	3 417	3 413	0 615
BA	245 543	237 543	241 543	4 615
CD	0 192	0 186	0 189	2 092
CA	9 309	9 417	9 363	12 408
CC	977 422	973 812	975 617	0 909
CC	2051 923	1963 509	2007 206	0 042
CD	2 356	2 426	2 391	1 693
CO	7 198	7 019	7 108	3 452
FO	10638 268	10408 513	10523 393	0 550
PS	138 105	132 788	135 446	0 130
MF	236 377	223 924	230 150	3 794
FL	4358 018	4612 199	4485 108	0 000
HI	85 441	86 117	85 779	1 920
RI	488 110	497 046	492 578	1 630
SA	0 910	10 000	5 455	0 073
AG	1 298	1 358	1 328	1 630
MA	41049 936	41846 467	41448 201	4 266
V	1 255	1 118	1 186	1 528
ZM	21 643	22 130	21 886	7 110
BI	24181 876	24639 590	24410 733	0 704
B	0 356	0 790	0 573	2 072
CA	44 488	45 615	45 051	3 328
LI	4135 956	4212 087	4174 021	2 352
P	13519 804	15225 278	14372 541	1 079
SI	309 096	303 039	306 067	20 498
SV	325 467	302 290	313 878	0 492
8	1456 660	1402 873	1429 766	1 399
SN	21 153	21 213	21 183	4 963
TI	3 022	2 917	2 969	3 605
ZF	0 981	1 013	0 997	2 078
TA	0 269	0 267	0 268	5 158
CA-1				

DATE	Analyte	SMCS 24		PREP BLK	Branch #1	Branch #2	Average	RPD	Spk 144	Spk 2	DETLDA	Ratio	mg	Mat B
11-13-11		4444	S Res	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	(ug/g)	S Res	(ug/g)	R/DL	Time	(g/g)
NCP-Acid	Al	3000	122.30	1.42E-01	J 6.71E-01	6.71E-01	6.71E-01	0.42	57.41	72.10	2.40E-00	194.47		
	Si	3000	71.90	1.77E-01	WT 5.19E-01	2.00E-01	3.59E-01	0.43	57.41	23.10	1.77E-01	2.01		
	As	3000	83.30	3.00E-00	WT 1.50E-00	1.19E-00	2.41E-00	3.08	57.41	60.10	3.00E-00	1.15		
	Ba	10000	92.70	3.00E-01	J 6.70E-01	6.67E-01	6.68E-01	0.40	114.81	98.60	3.00E-01	222.79		
	Ba	10000	97.10	1.00E-01	WT 1.20E-01	1.11E-01	1.17E-01	3.92	114.81	101.70	1.00E-01	1.17		
	Ca	10000	93.10	4.00E-01	J 4.70E-00	4.09E-00	4.40E-00	10.8	114.81	99.90	4.00E-01	10.99		
	Co	10000	112.70	3.11E-01	J 1.50E-01	1.49E-01	1.49E-01	0.34	114.81	114.30	4.40E-00	339.32	0.00	0.00
	Cr	3000	98.80	0.00E-01	J 2.01E-01	2.01E-01	2.04E-01	1.55	57.41	138.30	0.00E-01	2291.11	0.15	0.00
	Co	10000	93.60	0.00E-01	WT 1.10E-00	3.11E-00	3.11E-00	1.04	114.81	99.60	0.00E-01	3.91		
	Cu	3000	91.30	0.00E-01	J 1.63E-01	1.64E-01	1.64E-01	0.01	57.41	94.60	0.00E-01	41.12		
	Fe	3000	102.80	3.62E-00	J 1.74E-04	1.74E-04	1.73E-04	0.14	57.41	324.00	1.00E-00	17482.1	0.00	0.00
	Fe	3000	87.60	6.20E-00	J 2.01E-01	2.00E-01	2.01E-01	0.10	57.41	84.40	6.20E-00	32.37		
	Mg	3000	104.30	1.01E-00	J 1.07E-01	1.07E-01	1.01E-01	1.57	57.41	87.60	3.00E-01	1015.34		
	Mn	3000	91.10	2.00E-01	J 6.77E-01	6.43E-01	6.71E-01	1.73	57.41	103.40	2.00E-01	3334.1	0.00	0.00
	Ni	3000	96.00	1.70E-00	J 1.10E-03	1.09E-03	1.10E-03	0.60	57.41	101.30	1.70E-00	64.46		
	Ni	3000	107.70	1.12E-01	J 1.22E-01	1.21E-01	1.21E-01	0.80	57.41	82.30	1.12E-01	106.45	0.00	0.00
	Se	3000	88.60	7.60E-00	WT 1.20E-01	0.60E-00	1.03E-01	38.0	57.41	95.90	7.60E-00	1.34		
	Ag	3000	103.60	3.00E-01	J 4.31E-01	4.41E-01	4.43E-01	1.34	57.41	106.00	3.00E-01	84.64		
	Nb	10000	148.00	0.69E-01	J 3.30E-04	3.41E-04	3.30E-04	0.21	114.81	184.60	1.10E-00	11276.1	0.11	0.00
	V	10000	94.10	3.00E-01	J 1.42E-01	1.33E-01	1.39E-01	4.79	114.81	99.20	3.00E-01	27.74		
	Zn	10000	92.70	-3.51E-00	J 4.47E-01	4.34E-01	4.41E-01	2.45	114.81	101.10	3.00E-01	167.21		
	M	3000	48.80	7.30E-00	J 2.64E-04	2.64E-04	2.61E-04	0.64	57.41	230.70	7.30E-00	1630.80	0.41	0.01
	B	3000	101.30	1.23E-01	WT 2.94E-01	2.97E-01	2.94E-01	1.41	57.41	38.30	0.00E-01	48.99		
	Co	3000	97.30	1.01E-01	WT 3.57E-01	4.00E-01	3.78E-01	31.3	57.41	93.60	1.01E-01	3.75		
	La	3000	93.60	1.40E-00	J 4.67E-03	4.61E-03	4.64E-03	1.81	57.41	140.80	1.40E-00	3313.93	0.10	0.01
	P	10000	99.60	1.22E-01	J 9.73E-01	9.97E-01	9.84E-01	2.31	114.81	289.60	3.30E-00	1699.80	0.03	0.01
	Si	10000	171.20	7.41E-01	J 4.80E-01	3.71E-01	5.26E-01	10.1	114.81	-46.30	1.30E-00	403.91		
	Se	10000	92.80	3.00E-01	J 3.01E-02	3.01E-02	3.01E-02	2.30	114.81	94.90	3.00E-01	1815.00		
	S	3000	111.30	1.58E-01	J 1.14E-01	1.14E-01	1.14E-01	0.32	57.41	117.60	1.30E-00	422.80	0.11	0.00
	Se	3000	83.30	2.99E-00	WT 1.80E-00	1.81E-00	1.81E-00	0.55	57.41	90.30	1.80E-00	1.13		
	Ti	3000	92.90	4.00E-01	J 0.80E-00	0.80E-00	0.80E-00	2.24	57.41	84.10	4.00E-01	22.21		
	Zr	10000	90.30	0.00E-01	WT 9.20E-01	9.03E-01	9.13E-01	1.44	-	none	-	NA		

WMC-EP-0806

WMC-SD-WM-DP-024 ADDENDUM 2

REV. 0

11-13-11

[illegible]

DATE	ANALYST	LAB NO	SEC	TEST BLK	Result #1	Result #2	Average	STD	Spk	DET LMT	Ratio	
18-443	ICP-M. Laro	1000	88.70	1.77E-01	1.77E-01	1.77E-01	1.77E-01	27.23	1250	107.50	2.40E-00	EMA
		1000	88.50	3.00E-00	3.99E-00	3.99E-00	3.99E-00	0.00	2500	47.50	3.00E-00	1.00
		1000	89.00	3.00E-01	3.78E-01	3.78E-01	3.78E-01	61.60	2500	99.50	3.00E-01	1.12
		1000	101.50	1.01E-01	9.98E-02	9.98E-02	9.98E-02	0.00	2500	100.50	1.00E-01	1.00
		1000	101.80	4.00E-01	3.99E-01	3.99E-01	3.99E-01	0.00	2500	100.50	4.00E-01	1.00
		1000	97.50	3.94E-01	7.16E-01	6.64E-01	6.64E-01	113.60	2500	81.80	4.40E-00	13.10
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	1.59	107.00	99.50	9.00E-01	218.64
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1000	83.50	1.49E-01	6.69E-02	6.69E-02	6.69E-02	0.31	2500	102.40	1.50E-00	514.97
		1000	99.00	3.00E-01	2.38E-00	2.38E-00	2.38E-00	18.94	2500	99.50	3.00E-01	7.26
		1000	100.50	2.70E-00	1.07E-01	1.06E-01	1.06E-01	1.18	1250	103.00	2.70E-00	397.43
		1000	94.80	1.60E-00	1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.80	1.60E-00	1.00
		1000	98.80	1.13E-01	7.19E-02	7.19E-02	7.19E-02	3.81	1250	99.50	1.13E-01	63.54
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	0.00	1250	98.40	7.60E-00	1.00
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1000	83.50	1.49E-01	6.69E-02	6.69E-02	6.69E-02	0.31	2500	102.40	1.50E-00	514.97
		1000	99.00	3.00E-01	2.38E-00	2.38E-00	2.38E-00	18.94	2500	99.50	3.00E-01	7.26
		1000	100.50	2.70E-00	1.07E-01	1.06E-01	1.06E-01	1.18	1250	103.00	2.70E-00	397.43
		1000	94.80	1.60E-00	1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.80	1.60E-00	1.00
		1000	98.80	1.13E-01	7.19E-02	7.19E-02	7.19E-02	3.81	1250	99.50	1.13E-01	63.54
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	0.00	1250	98.40	7.60E-00	1.00
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1000	83.50	1.49E-01	6.69E-02	6.69E-02	6.69E-02	0.31	2500	102.40	1.50E-00	514.97
		1000	99.00	3.00E-01	2.38E-00	2.38E-00	2.38E-00	18.94	2500	99.50	3.00E-01	7.26
		1000	100.50	2.70E-00	1.07E-01	1.06E-01	1.06E-01	1.18	1250	103.00	2.70E-00	397.43
		1000	94.80	1.60E-00	1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.80	1.60E-00	1.00
		1000	98.80	1.13E-01	7.19E-02	7.19E-02	7.19E-02	3.81	1250	99.50	1.13E-01	63.54
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	0.00	1250	98.40	7.60E-00	1.00
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1000	83.50	1.49E-01	6.69E-02	6.69E-02	6.69E-02	0.31	2500	102.40	1.50E-00	514.97
		1000	99.00	3.00E-01	2.38E-00	2.38E-00	2.38E-00	18.94	2500	99.50	3.00E-01	7.26
		1000	100.50	2.70E-00	1.07E-01	1.06E-01	1.06E-01	1.18	1250	103.00	2.70E-00	397.43
		1000	94.80	1.60E-00	1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.80	1.60E-00	1.00
		1000	98.80	1.13E-01	7.19E-02	7.19E-02	7.19E-02	3.81	1250	99.50	1.13E-01	63.54
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	0.00	1250	98.40	7.60E-00	1.00
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1000	83.50	1.49E-01	6.69E-02	6.69E-02	6.69E-02	0.31	2500	102.40	1.50E-00	514.97
		1000	99.00	3.00E-01	2.38E-00	2.38E-00	2.38E-00	18.94	2500	99.50	3.00E-01	7.26
		1000	100.50	2.70E-00	1.07E-01	1.06E-01	1.06E-01	1.18	1250	103.00	2.70E-00	397.43
		1000	94.80	1.60E-00	1.60E-00	1.60E-00	1.60E-00	0.00	1250	93.80	1.60E-00	1.00
		1000	98.80	1.13E-01	7.19E-02	7.19E-02	7.19E-02	3.81	1250	99.50	1.13E-01	63.54
		1000	98.70	7.60E-00	7.58E-00	7.58E-00	7.58E-00	0.00	1250	98.40	7.60E-00	1.00
		1000	102.60	3.00E-01	4.99E-01	3.51E-01	3.51E-01	10.69	1250	102.00	3.00E-01	1.11
		1000	94.90	4.50E-01	3.09E-04	3.06E-04	3.06E-04	0.93	2500	80.40	3.10E-00	9926.17
		1000	100.90	3.00E-01	4.99E-01	4.99E-01	4.99E-01	0.00	2500	98.10	3.00E-01	1.00
		1000	101.50	3.13E-00	3.99E-01	3.99E-01	3.99E-01	0.00	2500	98.50	3.00E-01	1.00
		1000	101.70	7.50E-00	3.58E-02	3.58E-02	3.58E-02	21.03	1250	66.60	7.50E-00	50.64
		1000	99.10	8.06E-01	3.54E-00	3.54E-00	3.54E-00	0.00	1250	87.60	6.00E-01	9.23
		1000	101.60	1.01E-01	1.01E-01	1.01E-01	1.01E-01	0.00	1250	107.50	1.01E-01	1.00
		1000	97.50	1.40E-00	1.22E-01	1.38E-01	1.38E-01	24.07	1250	102.00	1.40E-00	9.87
		1000	97.50	3.34E-01	3.30E-01	3.30E-01	3.30E-01	1.62	2500	122.80	3.30E-00	913.61
		1										

[illegible]

T-111 Core 33 Composite 1

DATE	Analyte	EMCS 314		PREP BLK	Result #1	Result #2	Average	RPD	24 h44	24 h46	DET LDM	Ratio	max	Min
10/470		add (ug/l)	S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S	(ug/l)	S Rec	(ug/g)	R/DL	max	Min
KCP-Pulson	Al	3000	81 10	1.20E-01	J 4.81E-02	4.81E-02	4.81E-02	0.44	1250	94.00	1.20E-01	40.30		
	Se	3000	94 90	8.81E-01	UT 8.81E-01	1.29E-02	1.00E-02	37.39	1250	81.60	8.81E-01	1.23		
	As	3000	93 10	1.30E-01	UT 1.30E-01	1.30E-01	1.30E-01	0.20	2500	43.60	1.30E-01	1.09		
	Be	10000	93 70	1.30E-00	J 4.48E-01	6.80E-01	6.34E-01	1.89	2500	92.80	1.30E-00	43.41		
	Ba	10000	87 10	1.04E-00	UT 3.00E-01	4.99E-01	4.99E-01	0.20	2500	84.20	1.04E-00	0.47		
	Ca	10000	93 00	2.00E-00	UT 4.04E-00	6.74E-00	6.41E-00	10.49	2500	90.00	2.00E-00	3.21		
	Ca	10000	90 60	2.40E-02	J 1.91E-03	2.30E-03	2.22E-03	23.76	2500	87.20	2.40E-02	9.22		
	Cr	3000	91 70	4.30E-00	J 1.74E-03	1.81E-03	1.79E-03	2.90	1250	93.20	4.30E-00	396.84		
	Co	10000	92 40	4.00E-00	UT 1.30E-01	1.37E-01	1.33E-01	4.39	2500	90.10	4.00E-00	3.33		
	Cu	3000	93 20	2.25E-00	J 2.24E-01	2.16E-01	2.21E-01	4.87	1250	91.40	2.25E-00	9.84		
	Fe	3000	87 30	2.39E-01	J 1.37E-04	1.62E-04	1.39E-04	3.23	1250	824.90	2.39E-01	414.29		
	Pb	3000	93 40	3.10E-01	J 2.71E-02	2.62E-02	2.67E-02	3.69	1250	86.20	3.10E-01	8.63		
	Mg	3000	93 60	7.73E-00	J 2.18E-02	2.78E-02	2.48E-02	7.33	1250	91.30	7.73E-00	14.39		
	Mn	3000	90 20	1.33E-01	J 4.13E-03	6.29E-03	6.23E-03	3.21	1250	82.30	1.33E-01	401.84		
	Ni	3000	93 70	1.07E-02	J 9.09E-03	9.49E-03	9.29E-03	4.28	1250	84.10	1.07E-02	86.63		
	K	3000	97 60	3.60E-01	UT 3.60E-01	3.39E-01	3.39E-01	0.20	1250	-1097	3.60E-01	1.00		
	Na	3000	93 90	3.80E-01	UT 3.80E-01	3.79E-01	3.80E-01	0.20	1250	86.60	3.80E-01	1.00		
	Ag	3000	88 30	2.91E-00	J 4.00E-01	3.89E-01	3.93E-01	2.73	1250	97.90	2.91E-00	13.37		
	Hg	10000	87 30	6.34E-02	J 3.34E-04	2.41E-04	3.39E-04	1.49	2500	79.00	6.34E-02	30.39		
	V	10000	97 60	2.30E-00	J 1.89E-01	1.38E-01	1.33E-01	19.80	2500	91.70	2.30E-00	6.13		
	Zn	10000	94 40	1.37E-01	J 1.00E-02	1.10E-02	1.03E-02	9.39	2500	92.40	1.37E-01	2.67		
	M	3000	87 30	3.73E-01	J 2.61E-04	2.66E-04	2.63E-04	1.03	1250	33.30	3.73E-01	704.23		
	B	3000	81 30	3.83E-00	UT 4.32E-00	3.34E-00	4.84E-00	21.37	1250	78.80	3.83E-00	0.83		
	Co	3000	101 80	3.03E-01	UT 3.03E-01	3.04E-01	3.04E-01	0.20	1250	101.20	3.03E-01	1.00		
	Ia	10000	101 10	7.00E-00	J 4.43E-03	4.31E-03	4.31E-03	3.08	1250	94.30	7.00E-00	644.92		
	P	10000	93 80	8.73E-01	J 9.13E-03	8.99E-03	9.07E-03	1.81	2500	93.20	8.73E-01	103.69		
	M	10000	68 60	7.34E-01	J 3.39E-03	3.32E-03	3.46E-03	2.40	2500	68.30	7.34E-01	74.34	0.39	
	Se	10000	91 90	4.37E-00	J 2.44E-02	2.97E-02	2.91E-02	4.28	2500	91.90	4.37E-00	64.44		
	S	3000	96 40	1.33E-01	J 1.08E-03	1.08E-03	1.08E-03	0.18	1250	96.70	1.33E-01	79.93		
	Se	3000	93 30	8.00E-00	UT 8.00E-00	7.91E-00	7.99E-00	0.20	1250	92.30	8.00E-00	1.00		
	Ti	3000	92 40	2.00E-00	J 2.21E-01	2.23E-01	2.23E-01	1.16	1250	91.10	2.00E-00	11.16		
	Zr	—	—	4.00E-00	UT 1.00E-00	3.99E-00	4.00E-00	0.20	1250	—	4.00E-00	—		

WHC-EP-0806

WHC-SD-WH-DP-D24 ADDENDUM 2, REV 0

T-111 Core 33 Composite 1

			Water ICP/Acid ICP		Water ICP/Purion ICP		Purion ICP/Acid ICP	
Charge Bal	Tot Cat	3.36	Cr	0.108	Al	0.032	Al	1.073
	Tot An	2.28	Mn	0.004	Cr	0.125	Fe	0.012
	Ca/An	1.36	Si	1.269	Fe	0.006	Na	0.168
			B	0.928	Mn	0.004	Si	0.172
			Na	0.080	Si	0.009	Si	10.343
			Al	0.033	P	0.384	Ti	2.108
			K	0.166	Si	0.123	P	0.920
			P	0.537	Si	0.007	Si	0.934
Material Balance: outside model		0.93						

	Analyte	Std #1	PREP BLK	Result #1	Result #2	Average	RPD	Std Rec	DIET LDM	Ratio	moq	Mat Bal
		5 Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	%	(ug/g)	R/DL	Items	(g/g)
DATE 4-26-92	12.470											
Fluor	U	101.80	< 4.0E-2	J 3.34E-01	3.01E-01	3.18E-01	10.4	66.10	3.40E-02	0.34	0.027	0.00
	U as uCl/g					1.06E-01						
	Analyte	Std #1	Prep BLK	Result #1	Result #2	Average	RPD	Std Rec	DET LDM	Ratio	Rel.	
		5 Rec	(uCl/g)	(uCl/g)	(uCl/g)	(uCl/g)	%	%	(uCl/g)	R/DL	Cl Err	
DATE 4-26-92	12.470											
RAD-Flu.	TA	94.70	< 3.0E-3	J 3.76E-01	3.78E-01	3.77E-01	0.33	90.80	3.30E-04	711.32	1.40	
	TA/Pu+Am					3.19E-00						
DATE 4-26-92	12.470											
	TB	96.20	< 6.13E-2	9.32E-00	9.86E-00	9.39E-00	3.63	94.40	6.13E-02	133.93	1.20	
	TB/Cr-Si					9.11E-01						
DATE 4-27-92	12.470											
OEA	Cr-137	96.00	4.64E-04	1.13E-01	1.13E-01	1.14E-01	2.64	NA	3.70E-04	306.76	1.30	
	Eu-154	NA	< 1.02E-3	< 1.01E-3	< 1.11E-3	NA	NA	NA	1.20E-03	NA	NA	
	Eu-153	NA	< 6.83E-4	3.16E-03	3.97E-03	3.07E-03	6.20	NA	5.93E-04	3.13	47.0	
	Am-241	NA	< 1.37E-3	3.39E-01	3.93E-02	3.87E-02	4.13	NA	1.14E-03	33.93	0.80	
	Co-60	96.00	< 8.3E-4	< 7.0E-4	< 3.8E-4	NA	NA	NA	4.07E-04		NA	
DATE 5-4-92	12.470											
	PU238/40	93.70	< 3.6E-3	J 1.29E-01	1.39E-01	1.34E-01	2.46	89.20	3.30E-03	38.79	0.30	
	PU-236	93.70	NA	< 1.00E-3	< 1.03E-2	NA	NA	33.30	1.00E-01	NA	1.00	

T-111 Core 33 Composite 1

	Analyte	Std #1	Prop. Std	Result #1	Result #2	Average	R/PD	Std Rec	DET LDM	Ratio	Rel.
		g Rec	(uCVg)	(uCVg)	(uCVg)	(uCVg)	g	g	(uCVg)	B/DL	Cl. Err
Date: 3-19-92	18.470										
	Am-241	91.90	6.32E-03	J 3.94E-02	3.71E-02	3.82E-02	6.01	14.10	3.84E-03	6.34	10.0
	Am-244			none detected		NA	NA	6.00		NA	
	Am-241 (DEAP)/Am-241 (ALPHA)					1.01E-00					
DATE 4-24-92	18.470										
	HP-117	68.20	8.47E-01	U 3.24E-2	3.24E-2	NA	NA	74.30	3.24E-03	NA	3.60
DATE 4-28-92	18.470										
	TC-99	104.40	1.00E-02	J 1.14E-02	1.12E-02	1.14E-02	3.31	64.90	1.60E-03	2.48	3.60E-00
								69.00			
DATE 4-23-92	18.470										
	I-129	119.30	LOST	U 1.44E-2	1.13E-2	NA	NA	34.60	6.40E-03	NA	
								39.70			

WHC-EP-0806

WHC-SD-WM-OP-024 ADDENDUM 2, REV 0

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T-111 Core 33 Composite 1

	Analyte	Std #1	Prep Blk	Reach #1	Reach #2	Average	RPD	Spd Rec	DET LIM	Ratio	Cl Err	
		S Rec	(uCVg)	(uCVg)	(uCVg)	(uCVg)	%	%	(uCVg)	R/DL		
DATE 4-27-92	18.470											
	Se-90	92.20	< 1.5E-3	J 3.62E-00	3.67E-00	3.65E-00	1.37	94.80	2.13E-03	1693	0.40	
								94.70				
DATE 4-24-92	18.462											
	H-3	88.40	< 1.5E-4	< 1.5E-4 LA	< 1.5E-4	NA	NA	94.20	3.13E-04	NA	0.40	
DATE 4-24-92	18.462											
	C-14	88.90	< 1.5E-4	< 1.5E-4 W	< 1.5E-4	NA	NA	97.20	3.23E-04	NA	0.40	
DATE 4-29-92	18.470											
	Se-79	N/A	< 1.40E-4	LA < 1.26E-4	< 1.23E-4	NA	NA	89.30	1.40E-04	NA	0.30	
								91.20				
	Analyte	Std #1	PREP BLK	Reach #1	Reach #2	Average	RPD	Spd Rec	DET LIM	Ratio	ICLP Lim	Reache/Lim
		S Rec	(mg/L)	(mg/L)	(mg/L)	(mg/L)	%	%	(mg/L)	R/DL	(mg/L)	
Date:	18.611											
ICLP-ICP	Ag	102.00	3.00E-03	J 3.30E-02	3.47E-02	2.98E-02	32.30	23.80	3.00E-03	3.97	3.00	0.01
	Ba	94.00	3.00E-03	J 2.12E-02	3.02E-02	2.57E-02	33.03	19.40	3.00E-03	6.37	100.00	0.00
	Ca	93.80	4.00E-03	J 3.00E-02	3.30E-02	2.15E-02	34.33	103.30	4.00E-03	6.88	1.00	0.03
	Cl	101.60	3.00E-03	J 7.32E-00	7.37E-00	7.34E-00	0.33	80.10	9.00E-03	838.32	3.00	1.31
	Pb	94.30	6.20E-02	J 8.84E-01	4.72E-01	6.78E-01	60.73	-3.30	6.20E-02	10.93	3.00	0.14
	As	93.80	3.00E-02	J 1.30E-01	1.30E-01	1.30E-01	0.00	—	3.00E-02	3.00	3.00	0.03
	Se	93.30	7.80E-02	J 3.80E-01	3.80E-01	3.80E-01	0.00	—	7.80E-02	3.00	1.00	0.38
Date:	18.608											
ICLP-CVAA	Hg	100.20	< 0.100	W 3.10E-02	3.00E-02	3.03E-02	3.28	100.70	1.00E-02	3.03	0.20	0.13

WMC-SD-WM-DP-024 ADDENDUM 2, REV 0

WMC-EP-0806

[illegible]

T-111 Core 33 Composite 2

	Analyte	Sid #1	PREP BLK	Result #1	Result #2	Average	RPD	Sys Rec	DET LIM	Ratio	meq	Dist Bal
		S Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	S	S	(ug/g)	R/DL	lbs	(g/g)
	18 437											
	DSC	EXO	NA	NO EXO	NO EXO	NA	NA	NA	NA	NA		
	18 437			(R)	(R)	(R)						
	TGA	98.30	NA	8.30E-01	7.84E-01	8.08E-01	5.45	NA	NA	NA		
	18 422			(R)	(R)	(R)						
5 WATER		99.70	NA	J 7.64E-01	7.78E-01	7.71E-01	1.82	NA	NA	NA		0.77
	18 431											
HYAA	As	PHL										
	Se	PHL										
CYAA	Hg	101.40	<1.25E-1	J 1.15E-00	1.02E-00	1.08E-00	11.89	82.70	1.25E-01	0.08		
	18 446											
CN-DIR	CN	99.30	<3.00E0	UJ <4.61	<4.76	NA	NA	100.30	3.00E-00	NA	0.000	0
	CuNiFeCN6					NA					0.000	0

WHC-SD-WM-DP-024 ADDENDUM 2, REV 0

T-111 Core 33 Composite 2

DATE	Analysis	EALC3 Std		PHEP DLX	Result #1	Result #2	Average	RPD	Std add	Spike	DET LDI	Ratio
12-41		add'd	% Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	(ug/L)	% Rec	(ug/g)	R/DL
ICP-Water	Al	3000	98.70	2.40E-00	UT 1.02E-01	1.19E-01	1.10E-01	15.34	1250	107.30	2.40E-00	4.33
	Si	3000	100.10	1.77E-01	UT 1.77E-01	1.77E-01	1.77E-01	0.09	1250	107.50	1.77E-01	1.00
	As	3000	98.30	3.00E-00	UT 3.00E-00	2.99E-00	3.00E-00	0.10	2500	47.50	3.00E-00	1.00
	Ba	10000	99.00	3.00E-01	UT 3.01E-01	3.32E-01	3.16E-01	3.97	1500	99.50	3.00E-01	1.72
	Ba	10000	103.70	1.91E-01	UT 9.99E-02	9.91E-02	9.99E-02	0.10	2500	100.20	1.00E-01	1.00
	Ca	10000	101.90	4.00E-01	UT 4.00E-01	3.99E-01	3.99E-01	0.10	2500	100.20	4.00E-01	1.00
	Ca	10000	97.30	3.94E-01	UT 4.13E-01	9.33E-01	6.73E-01	77.04	2500	91.90	4.00E-00	15.33
	Cr	3000	101.30	9.00E-01	J 3.09E-02	2.17E-02	2.11E-02	1.71	1250	102.00	9.00E-01	234.18
	Co	10000	99.80	8.00E-01	UT 7.99E-01	8.50E-01	8.15E-01	6.17	2500	99.20	8.00E-01	1.01
	Cu	3000	96.40	4.00E-01	UT 4.00E-01	3.99E-01	3.99E-01	0.10	1250	97.50	4.00E-01	1.00
	Fe	3000	99.30	1.00E-00	J 1.38E-02	1.60E-02	1.39E-02	1.34	1250	98.50	1.00E-00	139.33
	Pb	3000	98.60	6.20E-00	UT 4.19E-00	6.18E-00	6.19E-00	0.10	1250	91.70	6.20E-00	1.00
	Mg	3000	101.90	1.78E-00	UT 3.77E-00	3.83E-00	3.83E-00	4.03	1250	101.60	3.00E-01	12.78
	Mn	3000	96.00	2.00E-01	J 3.42E-01	3.34E-01	3.38E-01	3.28	1250	95.10	2.00E-01	144.99
	Ni	3000	100.80	1.70E-00	UT 1.70E-00	1.70E-00	1.70E-00	0.10	1250	100.60	1.70E-00	1.00
	K	3000	98.80	1.12E-01	J 6.50E-02	6.47E-02	6.48E-02	0.30	1250	99.50	1.12E-01	37.90
	Se	3000	98.70	7.60E-00	UT 7.59E-00	7.88E-00	7.59E-00	0.10	1250	96.40	7.60E-00	1.00
	Ag	3000	102.60	3.00E-01	UT 4.99E-01	4.99E-01	4.99E-01	0.10	1250	102.00	3.00E-01	1.00
	Na	10000	96.90	4.50E-01	J 3.22E-04	3.18E-04	3.20E-04	0.64	2500	90.40	3.10E-00	10334.1
	V	10000	100.50	3.00E-01	UT 6.16E-01	1.31E-01	7.99E-01	30.69	2500	99.10	3.00E-01	1.60
	Zn	10000	101.30	3.33E-00	UT 3.00E-01	2.99E-01	3.00E-01	0.10	2500	98.20	3.00E-01	1.00
	Bi	3000	101.70	7.50E-00	J 2.47E-02	2.73E-02	2.70E-02	1.96	1250	64.60	7.50E-00	36.92
	B	3000	99.10	8.04E-01	J 4.04E-00	4.44E-00	4.25E-00	0.89	1250	97.60	8.00E-01	7.08
	Co	3000	103.60	1.91E-01	UT 1.91E-01	1.91E-01	1.91E-01	0.10	1250	107.50	1.91E-01	1.00
	La	--	--	1.60E-00	J 1.58E-01	1.57E-01	1.58E-01	0.10	1250	102.00	1.40E-00	11.23
	P	10000	97.20	3.80E-00	J 3.74E-03	3.64E-03	3.70E-03	1.34	2500	122.80	3.80E-00	983.21
	Si	10000	83.30	1.49E-01	J 6.11E-02	6.23E-02	6.20E-02	0.58	2500	102.40	1.30E-00	476.94
	Str	10000	99.00	3.00E-01	J 2.29E-00	2.39E-00	2.34E-00	4.24	2500	99.50	3.00E-01	7.79
	S	3000	100.30	2.70E-00	J 1.14E-03	1.14E-03	1.14E-03	0.41	1250	103.00	2.70E-00	422.81
	Sn	3000	96.80	1.60E-00	UT 1.60E-00	1.60E-00	1.60E-00	0.10	1250	91.80	1.60E-00	1.00
	Ti	3000	107.30	4.00E-01	UT 4.00E-01	3.99E-01	3.99E-01	0.10	1250	100.00	4.00E-01	1.00
	Zr	10000	103.40	3.00E-01	UT 7.99E-01	7.94E-01	7.99E-01	0.10	1250	--	--	--

MHC-SD-WH-DP-024 ADDENDUM 2, REV 0

MHC-EP-0806

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T-111 Core 33 Composite 2

T-111 Core 33 Composite 2

DATE:	Analysis	INCS Std		PREP BLK		Result #1	Result #2	Average	RPD	Std Std	Spike	DET LDM	Ratio	...
11 478		Std (ug/l)	S Rec	(ug/g)		(ug/g)	(ug/g)	(ug/g)	%	(ug/l)	S Rec	(ug/g)	R/DL	...
ICP Fusion	Al	5000	91 10	1.20E-01	J	4.39E-02	4.39E-02	4.39E-02	0.07	1250	94.00	1.20E-01	38.25	
	Sb	5000	94 90	8.85E-01	WJ	8.85E-01	8.85E-01	8.85E-01	0.20	1250	85.60	8.85E-01	1.00	
	As	5000	93 10	1.30E-01	WJ	1.30E-01	1.30E-01	1.30E-01	0.20	2500	45.60	1.30E-01	1.00	
	Ba	10000	93 70	1.30E-00	J	7.30E-01	7.43E-01	7.37E-01	2.01	2500	92.80	1.30E-00	49.14	
	Ba	10000	97 10	1.06E-00	WJ	4.99E-01	5.00E-01	4.99E-01	0.20	2500	94.20	1.06E-00	0.47	
	Cd	10000	93 00	2.00E-00	WJ	7.48E-00	8.04E-00	7.77E-00	8.65	2500	90.00	2.00E-00	3.59	
	Ca	10000	90 60	2.40E-02	J	1.92E-03	2.18E-03	2.05E-03	12.7	2500	87.20	2.40E-02	0.34	
	Cu	5000	93 70	4.50E-00	J	1.82E-03	1.82E-03	1.82E-03	0.16	1250	93.20	4.50E-00	404.16	
	Cu	10000	92 40	4.00E-00	WJ	1.48E-01	8.90E-00	1.18E-01	49.3	2500	90.10	4.00E-00	2.96	
	Cu	5000	92 20	2.25E-00	J	2.61E-01	2.30E-01	2.46E-01	12.6	1250	91.40	2.25E-00	10.94	
	Fe	5000	92 30	2.39E-01	J	1.61E-04	1.61E-04	1.61E-04	0.15	1250	121.90	2.39E-01	621.74	
	Pb	5000	93 40	3.10E-01	J	2.67E-02	2.72E-02	2.69E-02	1.71	1250	86.20	3.10E-01	8.69	
	Mg	5000	93 40	7.75E-00	J	2.69E-02	2.74E-02	2.72E-02	1.84	1250	91.30	7.75E-00	35.06	
	Mn	5000	90 20	1.55E-01	J	6.39E-03	6.39E-03	6.39E-03	0.011	1250	82.50	1.55E-01	423.63	
	Ni	5000	93 70	1.07E-02	J	1.43E-04	9.81E-03	1.31E-04	37.2	1250	84.10	1.07E-02	182.41	
	K	5000	97 60	3.60E-03	WJ	3.39E-01	3.60E-01	3.39E-01	0.20	1250	-1097	3.60E-01	1.00	
	Se	5000	93 90	3.80E-01	WJ	3.79E-01	3.80E-01	3.80E-01	0.20	1250	86.60	3.80E-01	1.00	
	Ag	5000	88 90	2.91E-00	J	3.74E-01	3.69E-01	3.71E-01	1.47	1250	97.90	2.91E-00	12.77	
	Na	10000	92 30	6.34E-02	J	3.32E-04	3.31E-04	3.32E-04	0.33	2500	79.00	6.34E-02	35.47	
	V	10000	92 60	2.30E-00	J	1.31E-01	1.42E-01	1.47E-01	5.94	2500	91.70	2.30E-00	3.87	
	Zn	10000	94 40	1.37E-01	J	1.19E-02	1.02E-02	1.10E-02	15.4	2500	92.40	1.37E-01	0.04	
	Bi	5000	97 30	3.75E-01	J	2.61E-04	2.73E-04	2.67E-04	4.52	1250	33.30	3.75E-01	711.88	
	B	5000	81 30	3.85E-00	WJ	3.10E-00	4.58E-00	4.84E-00	10.9	1250	78.80	3.85E-00	0.83	
	Ce	5000	101 80	3.05E-01	WJ	3.04E-01	3.05E-01	3.04E-01	0.20	1250	103.20	3.05E-01	1.00	
	La	5000	101 10	7.00E-00	J	4.78E-03	4.84E-03	4.81E-03	1.36	1250	94.50	7.00E-00	687.17	
	P	10000	93 80	8.75E-01	J	9.91E-03	9.91E-03	9.91E-03	0.01	2500	93.20	8.75E-01	113.30	
	Si	10000	99 60	7.39E-01	J	3.41E-03	3.41E-03	3.41E-03	0.04	2500	68.50	7.34E-01	73.69	0.91
	Si	10000	93 90	4.37E-00	J	3.36E-02	2.98E-02	3.17E-02	11.9	2500	91.90	4.37E-00	72.45	
	S	5000	96 40	1.35E-01	J	1.16E-03	1.16E-03	1.16E-03	0.52	1250	96.70	1.35E-01	83.79	
	Se	5000	93 30	8.00E-00	WJ	7.91E-00	8.00E-00	7.99E-00	0.20	1250	92.30	8.00E-00	1.00	
	Ti	5000	92 40	2.00E-00	J	2.31E-01	2.31E-01	2.41E-01	8.62	1250	91.10	2.00E-00	12.05	
	Zr	—	—	4.00E-00	WJ	3.99E-00	4.00E-00	4.00E-00	0.20	—	—	4.00E-00	—	

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WMC-BD+WN-CP-b24 ADDENDUM 2, REV 0

T-111 Core 33 Composite 2

			Waters K/P/Acid ICP		Waters ICP/Fusion ICP		Fusion ICP/Acid ICP	
Charge Bal	Tot Cat	3.38	Cs	0.10	Al	0.024	Al	1.13
	Tot An	2.38	Mn	0.01	Cr	0.116	Pb	0.93
	Ca/An	1.39	Ni	1.37	Pb	0.018	Ni	0.97
			0	0.04	Mn	0.003	Bi	0.04
			Na	0.98	Bi	0.018	Bi	11.74
			Al	0.03	P	0.373	Ti	1.73
			K	0.04	Si	0.113	P	0.97
			P	0.30	Se	0.007	Se	0.95
Material Balance: oxide model								

Date: 4/27/92	Analyte	Std #1	FILED BLE	Result #1	Result #2	Average	RPD	Std Dev	DET LIM	Ratio	mg	Mat Bal
18.471		8 Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	%	%	(ug/g)	R/DL	lbs	(g/g)
Fluor	U	101.00	< 40E-3	1.02E-03	2.07E-03	1.93E-03	12.83	66.10	3.40E-02	3.72	0.014	0.002
	U as uCVg					6.47E-04						
	Analyte	Std #1	FILED BLE	Result #1	Result #2	Average	RPD	Std Dev	DET LIM	Ratio	mg	Mat Bal
		8 Rec	(uCV/g)	(uCV/g)	(uCV/g)	(uCV/g)	%	%	(uCV/g)	R/DL	Cl Ent	
DATE: 4-26-92	18.471											
TAAD-Fue.	TA	04.70	< 3.0E-3	3.97E-01	3.97E-01	3.97E-01	0.00	00.00	3.30E-04	749.04	1.400	
	TA/Pr-Am					2.03E-00						
DATE: 4-26-92	18.471											
	TB	04.70	< 6.13E-2	6.93E-00	6.93E-00	6.93E-00	1.33	04.40	6.13E-02	144.31	1.300	
	TB/Cs-Sr					9.14E-01						
DATE: 4-27-92	18.471											
OEa	Cs-137	04.00	4.64E-02	1.04E-01	1.03E-01	1.03E-01	0.97	NA	3.70E-04	279.73	1.600	
	Eu-154	NA	< 1.01E-3	< 9.64E-4	< 1.03E-3	NA	NA	NA	1.20E-03	NA	NA	
	Eu-155	NA	< 6.83E-4	< 1.49E-3	< 1.49E-3	NA	NA	NA	3.91E-04	NA	NA	
	Am-241	NA	< 1.39E-3	4.61E-02	4.24E-02	4.43E-02	0.34	NA	1.14E-04	314.16	7.800	
	Cs-144	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	
	Cs-60	04.00	< 1.1E-4	< 4.9E-4	< 3.9E-4	NA	NA	NA	4.97E-04	NA	NA	
DATE: 5-6-92	18.471											
	PU239/40	76.30	< 3.4E-3	1.42E-01	1.33E-01	1.47E-01	7.46	04.80	3.30E-03	42.14	0.300	
	PU-238	76.30	< 3.4E-3	< 1.04E-2	< 1.13E-2	NA	NA	76.60	1.60E-03	NA	0.900	

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WHC-SD-NM-DP-024 ADDENDUM 2, REV 0

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T-111 Core 33 Composite 2

	Analyte	Sid #1	PREP BLE	Result #1	Result #2	Average	RPD	Sys Rec	DET LDM	Ratio	Relative
Date: 3/18/92	18.471	8 Rec	(ug/g)	(ug/g)	(ug/g)	(ug/g)	11	8	(ug/g)	B/DL	IS Cl. Err
	Am-241	91.90	6.32E-03	J 5.11E-02	4.44E-02	4.78E-02	14.03	19.00	6.00E-03	7.104	0.70
	Am-241			none detected		NA	NA	18.70		NA	
	Am-241 (DEAP)/Am-241 (ALPHA)					9.97E-03					
DATE: 4-24-92	18.471										
	HP-237	68.20	8.47E-01	U.S. < 24E-1	< 24E-1	NA	NA	69.20	3.40E-02	NA	6.40
Date:	18.										
	TC-99	106.40	1.00E-02	J 1.03E-02	1.03E-02	1.04E-02	1.92	67.00	4.60E-03	2.26	3.70
								68.40			
DATE: 4-25-92	18.471										
	I-129	119.30	LOST	U.S. < 17E-1	< 1.7E-1	NA	NA	119.70	6.40E-03	NA	
								11.80			
DATE: 4-27-92	18.471										
	Se-90	92.20	< 13E-1	J 3.48E-00	3.17E-00	3.43E-00	3.21	93.40	2.15E-03	1393.0	0.40
								94.10			
DATE: 4-28-92	18.463										
	H-3	81.40	< 13E-1	U.S. < 13E-1	< 13E-1	NA	NA	84.20	3.15E-04	NA	6.30
DATE: 4-29-92	18.463										
	C-14	88.90	< 23E-1	U.S. < 23E-1	< 23E-1	NA	NA	87.80	2.25E-04	NA	4.20
DATE: 4-29-92	18.471										
	Se-79	N/A	< 1.40E-1	U < 1.24E-1	< 1.24E-1	NA	NA	89.30	1.40E-04	NA	4.30
								87.50			

There were no TCLP analysis required for this core composite sample.

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ADDITIONAL DATA SUBMITTED ON 05/18/94	96
Differential Scanning Calorimetry Analysis J-1668-J1672	97
Thermal Gravimetric Analysis J-1668-J1672	117

This report consists of pages 1 through 135, plus 3.1-3.3, 4.1, 5.1, 9.1-9.30, 23.1-23.2 and 32.1.

188.5 J/g calculated dry (wt. basis) value compared to average May exotherm values of 280.9 J/g on a calculated dry (wt. basis) value. The exotherm trace obtained in May is less well resolved than the January scan. The exotherms are very broad and not well separated from the water endotherms, so the baselines have been conservatively estimated.

Comparison of the current 222-S data (May 1994), and the original January 1992 data obtained for Core 33 segments 1 and 2 is quite interesting. The original data (Jan. 1992) was obtained on a wet sample under air. The January 1994 and current data are on a dried sample as previously described. The wet sample (Jan. 1992), Core 33 segment 1 yielded an average value of 1151 J/g dry (wt. basis), and 2848.5 J/g dry (wt. basis) for segment 2. Both of these values are considerably different from the values obtained on the dried sample either in January or May 1994.

Samples from Core 33 segment 2 were also run by PNL in January 1994, reference 6. Results for Core 2 samples run under nitrogen purge were 942 J/g dry (wt. basis) and 1011 J/g dry (wt. basis) for an average value of 976.5 J/g dry. Sample preparation for the January 1994 samples run by both labs were similar. The PNL sample was vacuum dried at 60 degrees C for 24 hours. At 222-S the sample was vacuum dried (35 torr) at 60 degrees C to a constant weight. The two laboratories used different heating rates for the DSC. The 222-S lab used 10 C per minute, while PNL ran their sample at 5 C per minute. This difference should not effect the magnitude of the exotherm, but may cause a shift in the curve. The instruments used by the laboratories were also different. The instrument used at 222-S is a Mettler M3 balance DSC Model 20 TG-50 , and at PNL a Perkin Elmer Model 2 was used.

Tank 241-T-111 Limited Analysis of Core 33 Segment 2Introduction

The analyses in this package were performed by the Westinghouse Hanford Company (WHC), under the guidance provided by the Analytical Integration Characterization Program (AICP), refer to references 1, 2, and 3. The analyses presented in this report provides additional information on the energetics previously observed for Tank 241-T-111, reference 4. There were no exotherms that exceeded the safety criteria.

Sample preparation and analyses were performed by the Analytical Services of WHC. Core 33 segment 2 was placed in an oven on 1/10/94 at 1500 with an initial weight of 28.64 grams (gms) and removed on 1/12/94 at 0735 and was stabilized in a hood for 1 hour, the final weight of the sample was 6.82 gms. The dried sample was stored in archive and was retrieved in May to perform this body of work. The specific analyses requested included Differential Scanning Calorimetry (DSC), which was performed under a nitrogen atmosphere on the sample previously dried under vacuum (35mm of Hg) at 60 degrees C. In addition, Thermogravimetric Analysis (TGA) were performed on the dried archive sample. The analyses were performed to the quality assurance protocols reference 7.

Data Evaluation of Tank 241-T-111

Thermogravimetric analysis on the dried samples yielded an average value of 11.2% weight loss interpreted to be water loss. Differential Scanning Calorimetry of 5-10 mg. and 10-20 mg. sample sizes under nitrogen purge were performed. This analysis (May 1994) of Core 33 segment 2 showed consistent low broad exotherms, with no significant differences caused by sample sizes. These results are consistent with previous work performed on Core 33 segment 2 in January 1994, reference 5. The average DSC exotherm value from January was

References:

1. Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction on Centrifugation of Tank 241-T-111 Samples," dated April 22, 1994.
7E720-94-119 Westinghouse Hanford Company, Richland Wa.
2. Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction for Tank 241-T-111 Archive Samples," dated April 29, 1994.
7E720-94-120 Westinghouse Hanford Company, Richland Wa.
3. Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Revised Letter of Instruction for Tank 241-T-111 Archive Samples." dated May 2, 1994.
Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction for Tank 241-T-111 Archive Samples," dated April 29, 1994.
7E720-94-122 Westinghouse Hanford Company, Richland Wa.
4. WHC-SD-WM-DP-024, Rev 0. Tank 241-T-111 Core 31 and Core 33.
5. WHC-SD-WM-DP-058, Rev 0. Tank 241-T-111 Reanalyses of T-111 limited Analysis.
6. Pacific Northwest Laboratories, Tank Waste Characterization Project.
Tank 241-T-111 Core 33 Data Report Rev. 0. January 10, 1994.
Pacific Northwest Laboratories, Richland Wa.
7. WHC-SD-WM-PLN-047, Appendix A, Core Sample Characterization Quality Assurance Project Plan.

Summary Table for Tank 241 - T-111 Core 33 Segment 2, dried archive sample.

Sample Prep.	Analyte	Safety Criteria J/g dry	Std. #1 %	Result #1 J/g	Result #2 J/g	Average J/g	RPD %
			J1667-5511	J1668-5711	J1668-5711		
Direct	DSC	>522 J/g dry	96.0	251.2	269.0	260.1	6.84
			J1667-5511	J1669-5711	J1669-5811		
Direct	DSC	>522 J/g dry	96.0	309.2	287.5	298.4	7.27
			J1667-5511	J1670-5711	1670-5811		
Direct	DSC	>522 J/g dry	96.0	180.2	187.1	183.7	3.76
			J1667-5511	J1671-5711	J1671-5811		
Direct	DSC	>522 J/g dry	96.0	162.7	175.3	168.8	7.46
			J1667-5511	J1672-5711	1672-5811		
Direct	DSC	>522 J/g dry	96.0	336.2	335.9	336.0	0.09
			%	%	%	Average %	
			J1667-5512	J1668-5712	J1668-5812		
Direct	TGA		99.1	11.68	10.06	10.9	14.86
			J1667-5512	J1669-5712	J1669-5812		
Direct	TGA		99.1	10.66	9.90	10.3	7.38
			J1667-5512	J1670-5712	1670-5812		
Direct	TGA		99.1	11.35	11.99	11.7	5.47
			J1667-5512	J1671-5712	J1671-5812		
Direct	TGA		99.1	12.07	12.08	12.075	0.08
			J1667-5512	J1672-5712	J1672-5812		
Direct	TGA		99.1	11.21	11.10	11.15	0.99

WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-0

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Hanford Company

WHC-SD-WM-DP-058

REV. 0-KB 11/24/94

Internal
Memo

From: Analytical Integration

7E720-94-119

Phone: 373-3579 R2-18

Date: April 29, 1994

Subject: LETTER OF INSTRUCTION FOR TANK 241-T-111 ARCHIVE SAMPLES

To: J. G. Kristofzski T6-06

cc: H. Babad	R2-78	K. L. Kocher	T6-06
G. S. Barney	T5-12	R. P. Marshall	T6-14
D. B. Bechtold	T6-09	L. M. Sasaki	R2-12
C. DeFigh-Price	R2-31	P. Segall	H4-19
D. B. Engelman	R1-49	B. C. Simpson	R2-12
J. M. Grigsby	H4-62	D. A. Turner	R2-78
C. S. Haller	R2-12	for T. E. Whelan HKA	S1-57
J. M. Kier	T3-01	LMS File/LB 4/29/94	

Reference: Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction on Centrifugation of Tank 241-T-111 Samples," dated April 22, 1994.

This letter of instruction provides direction to the 222-S Laboratory for the performance of differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) tests on archived material from single-shell tank 241-T-111. This letter also cancels the centrifugation and moisture measurements requested in the previous letter of instruction (Reference). The previously requested tests were determined not to be an effective means of addressing the U.S. Department of Energy, Headquarters' concerns regarding the safety of pumping the tank. Alternative testing to estimate the moisture that will remain in the waste after pumping is being considered and may be requested in the future. However, it has been determined that additional information on the waste energetics is necessary to evaluate the safety of pumping the tank.

Purpose: Perform additional DSC analysis and TGA on tank 241-T-111 archive sample to more accurately measure the exotherm observed in the sample.

Safety Considerations: Radioactive materials will be used in these tests, therefore all laboratory procedures applicable to radiological control will be adhered to in conjunction with as low as reasonable achievable (ALARA) practices dealing with hazardous materials in the preparation and handling of these samples.

Work Scope:

1. The sample to be analyzed in the archived sample from tank 241-T-111, core 33, segment 2 which had been dried, but never used, for adiabatic calorimetry analysis. Homogenize the sample to ensure that it is well mixed.

J. G. Kristofzski
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April 29, 1994

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2K2

2. Obtain three aliquots from the sample and perform DSC and TGA analyses in duplicate on each aliquot. The sample size for the DSC analysis should be 5 to 10 milligrams. Perform the DSC under a nitrogen atmosphere.
3. Obtain two aliquots from the sample and perform DSC and TGA analyses in duplicate on each aliquot. The sample size for the DSC analysis should be twice the amount used in step 2 above (i.e., 10 to 20 milligrams). Perform the DSC under a nitrogen atmosphere.

Quality Assurance: If the sample and duplicate for a given aliquot do not agree within 25 calories/gram, rerun the DSC using a sample from the same aliquot. Approval designator Q has been established for this work; samples and the indicated number of duplicates shall be run. The DSC shall be performed in accordance with procedure number LA-514-113 and the TGA shall be performed in accordance with procedure number LA-560-112. The requirements of WHC-SD-CP-QAPP (Quality Assurance Project Plan for the Analysis of Highly Radioactive Mixed Waste Samples in Support of Environmental Activities on the Hanford Site) shall be implemented as applicable. The data shall be reviewed to ensure that all quality assurance/quality control requirements were met.

Data Evaluation: The 222-S Laboratory shall evaluate the results of the analyses against the DSC analyses performed previously on the tank T-111 samples that show exotherms in excess of 200 calories/gram (both cores 31 and 33) at both the 222-S and 325 laboratories. The evaluation should include a best estimate of the sample energetics along with uncertainties and the rationale behind the estimate.

Reporting Requirements: A letter presenting the results of the measurements will be the final deliverable of this task. The letter shall include a summary table of the results and a narrative. Advance notification of results via cc:mail is requested for representatives from the Characterization Program (D. R. Bratzel), Characterization Support (B. C. Simpson), Waste Tank Safety (H. Babad), Waste Tank Stabilization (D. B. Engelman), and TWRS Safety Analysis and Engineering (J. M. Grigsby). All analytical results shall be reported by May 3, 1994, the data evaluation shall be reported by May 5, 1994, and the letter report shall be issued by May 9, 1994.

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J. G. Kristofzski
Page 3
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7E720-94-119

Funding for this task is provided under task package control number N54D2.
A cost estimate should be provided to the Characterization Program by
May 3, 1994.

If there are any questions or comments regarding this letter, please contact
L. M. Sasaki at 373-1027 or B. C. Simpson at 373-5915.

DRB

D. R. Bratzel, Manager
Analytical Integration
Characterization Program

mjg

Westinghouse
Hanford Company

WHC-SD-WM-DP-058

REV. 0-XB

Internal
Memo

From: Analytical Integration 7E720-94-120
Phone: 373-3579 R2-18
Date: May 2, 1994
Subject: REVISED LETTER OF INSTRUCTION FOR TANK 241-T-111 ARCHIVE SAMPLES

To:	J. G. Kristofzski	T6-06		
cc:	H. Babad	R2-78	K. L. Kocher	T6-06
	G. S. Barney	T5-12	R. P. Marshall	T6-14
	D. B. Bechtold	T6-09	L. M. Sasaki	R2-12
	C. DeFigh-Price	R2-31	P. Segall	H4-19
	D. B. Engelman	R1-49	B. C. Simpson	R2-12
	J. M. Grigsby	H4-62	D. A. Turner	R2-78
	C. S. Haller	R2-12	T. E. Whelan	S1-57
	J. M. Kier	T3-01	LMS File/LB	

Reference: Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction for Tank 241-T-111 Archive Samples," dated April 29, 1994.

This letter of instruction (LOI) provides a revision to the referenced LOI. This revision expands the work scope to include additional differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) tests on wet (as received) archive sample from tank 241-T-111, core 33, segment 2 and revises the due dates for the reporting of results. All other requirements of the referenced LOI remain the same.

Additional Work Scope:

1. The additional sample to be analyzed is the 20 gram of wet sample from the tank 241-T-111, core 33, segment 2 archived sample. This sample is being shipped from the Pacific Northwest Laboratory (PNL) 325 Building to the 222-S Laboratory for analysis. PNL is not able to analyze the sample at this time due to the stand down at the laboratory. Homogenize the sample to ensure that it is well mixed.
2. Obtain three aliquots from the sample and perform DSC and TGA analyses in duplicate on each aliquot. The sample size for the DSC analysis should be 5 to 10 milligrams. Perform the DSC under a nitrogen atmosphere.
3. Obtain two aliquots from the sample and perform DSC and TGA analyses in duplicate on each aliquot. The sample size for the DSC analysis on the wet sample should be five times the amount used in step 2 above (i.e., 25 to 50 milligrams). Perform the DSC under a nitrogen atmosphere.

Change to Reporting Due Dates:

Analytical results and preliminary data evaluation on the dried archived sample shall be reported by May 6, 1994. Final evaluation of both dry and wet sample results, in the form of a letter report, shall be made within four full working days after receipt of the sample from PNL.

J. G. Kristofzski
Page 2
May 2, 1994

WHC-SD-WM-DP-058

7E720-94-120

REV. O-KB

Law 7/24/94

If there are any questions or comments regarding this letter, please contact
L. M. Sasaki at 373-1027 or B. C. Simpson at 373-5915.

L. M. Sasaki
for D. R. Bratzel, Manager
Analytical Integration
Characterization Program

Westinghouse
Hanford Company

WHC-SD-WM-DP-058

REV. O-B

Internal
Memo

From: Analytical Integration 7E720-94-122
Phone: 373-3579 R2-18
Date: May 12, 1994
Subject: LETTER OF INSTRUCTION FOR TANK 241-T-111 ARCHIVE SAMPLES -
REVISION 2

To: J. G. Kristofzski T6-06

cc: H. Babad	R2-78	K. L. Kocher	T6-06
G. S. Barney	T5-12	R. P. Marshall	T6-14
D. B. Bechtold	T6-09	L. M. Sasaki	R2-12
C. DeFigh-Price	R2-31	P. Segall	H4-19
D. B. Engelman	R1-49	B. C. Simpson	R2-12
J. M. Grigsby	H4-62	D. A. Turner	R2-78
C. S. Haller	R2-12	T. E. Whelan	S1-57
J. M. Kier	T3-01	LMS File/LB	

References: (1) Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Revised Letter of Instruction for Tank 241-T-111 Archive Samples," dated May 2, 1994.

(2) Internal Memo, D. R. Bratzel to J. G. Kristofzski, "Letter of Instruction for Tank 241-T-111 Archive Samples," dated April 29, 1994.

This letter of instruction (LOI) provides a second revision to the referenced LOIs. This revision eliminates the additional work scope requested in Reference 1. It has been determined that additional differential scanning calorimetry and thermal gravimetric analysis tests on wet (as received) archive sample from tank 241-T-111, core 33, segment 2 are not needed at this time. The final letter report should be issued by May 19, 1994.

If there are any questions or comments regarding this letter, please contact L. M. Sasaki at 373-1027 or B. C. Simpson at 373-5915.

DRB *atj*

D. R. Bratzel, Manager
Analytical Integration
Characterization Program

pkc

TO: JG Kristofzski, JM Frye, KL Kocher
 FROM: TL Welsh, RD Cromar, R Jeppson
 DATE: May 6, 1994
 SUBJECT: Statistical Analysis of Core Segments

WHC-SD-WM-DP-058

REV. 0-3

Performing a one-way analysis of variance (ANOVA) on segment two (core 33 only), we found that the standard deviation of segment two is 211.631 J/g dry wt. The ANOVA also leads us to conclude that some of the means are different. A multiple range comparison test showed that the means of the 1993 T-111 data and the present data are not statistically different, with a significance level of 0.05. These two means differ from the mean of the 1993 PNL data and also from the mean of the original data. The 1993 PNL mean and the mean of the original data also differ from each other.

Performing a one-way analysis of variance on segment one (core 33 only), the standard deviation of segment one is 169.621 J/g dry wt. The ANOVA leads us to conclude that the mean of the original data and the mean of the 1993 T-111 data are different. Using the error from segment two to test segment one, the means are also statistically different, with a significance level of 0.05.

Summary Statistics for Core Samples
 (in J/g dry wt)

	Confidence Interval		Mean	Standard Deviation	RSD
	L.L.	U.L.			
Core 33, 1994	*	*	280.9	81.29	0.2894
Segment 1	441.50	3088.17	1764.83	532.67	0.3018
Segment 2	-164.55	2671.11	1253.28	1142.06	0.9113
Segment 3	-14518.56	17932.56	1707	1805.95	1.0580

Segment 1

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REV. 0-B

Analysis of variance

Source of variation level	Sum of Squares	d.f.	Mean square	F-ratio	Sig.
Between groups	911070.25	1	911070.25	31.666	.0302
Within groups	57542.50	2	28771.25		
Total (corrected)	968612.75	3			

0 missing value(s) have been excluded.

adjusted F = 20.34
p-value = .00071

Segment 2

REV. 0-6

Analysis of variance

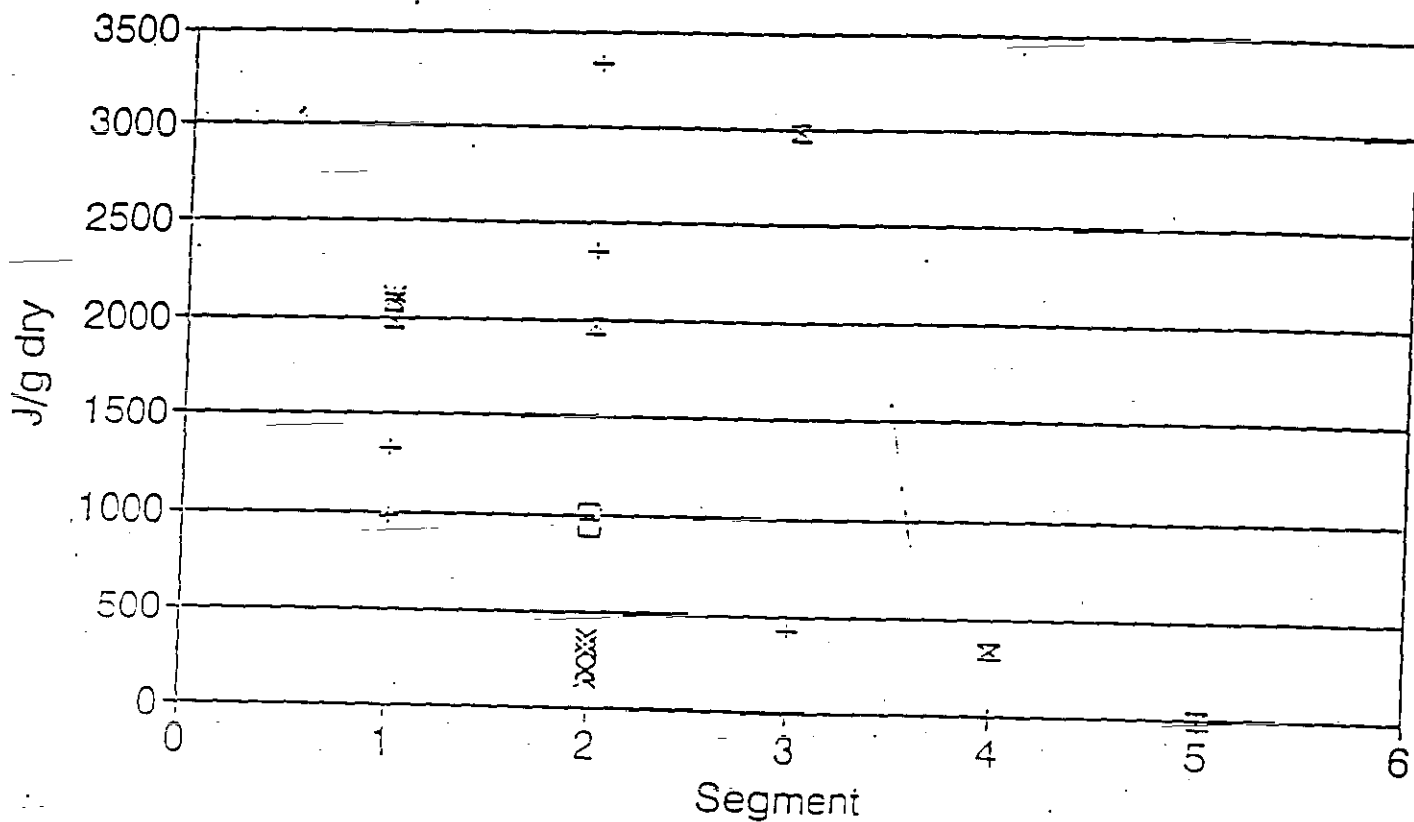
Source of variation level	Sum of Squares	d.f.	Mean square	F-ratio	Sig.
Between groups	11656441	3	3885480.2	86.753	.0000
Within groups	537452	12	44787.7		
Total (corrected)	12193893	15			

0 missing value(s) have been excluded.

WHC-SD-WM-DP-058

REV. 0-13

T-111 DSC



⌘ Core 31 (1991)	+ Core 33 (1991)	⌘ Core 33 (1993)
□ Core 33 (1993) P	× Core 33 (1994)	

Preliminary Data for re-analysis on tank T-111 Core 33 Segment 2, May 2, 1994
 All data is reported on a archive sample which was vacuum dried 60 degrees C. on 12/93. The analyses were performed under a nitrogen purge. This preliminary data fulfills the requirements per the letter or instruction (LOI) 7E720-94-119 and the revised LOI 7E720-94-120. The DSC values are not corrected and do not take into account the water content of the samples.

Laboratory Id. number	Sample #	Analysis	Sample Size (mg)	Result 1 J/g	Result 2 J/g
J1668-5711 J1668-5811	Sample 1 Sample 1 dup	DSC	16.957 13.267	251.2	269.0
J1669-5711 J1669-5811	Sample 2 Sample 2 dup	DSC	12.596 18.182	309.2	287.5
J1670-5711 J1670-5811	Sample 3 Sample 3 dup	DSC	5.897 6.179	180.2	187.1
J1671-5711 J1671-5811	Sample 4 Sample 4 dup	DSC	6.695 7.023	162.7	175.3
J1672-5711 J1672-5811	Sample 5 Sample 5 dup	DSC	7.740 6.154	336.3	335.9
Laboratory Id. number	Sample #	Analysis	Sample Size (mg)	Result 1 (% H2O)	Result 2 (% H2O)
J1668-5712 J1668-5812	Sample 1 Sample 1 dup	TGA	11.928 15.604	11.68	10.06
J1669-5712 J1669-5812	Sample 2 Sample 2 dup	TGA	17.548 19.760	10.66	9.90
J1670-5712 J1670-5812	Sample 3 Sample 3 dup	TGA	6.338 8.043	11.35	11.99
J1671-5712 J1671-5812	Sample 4 Sample 4 dup	TGA	7.786 5.750	12.07	12.08
J1672-5712 J1672-5812	Sample 5 Sample 5 dup	TGA	7.101 9.313	11.21	11.10

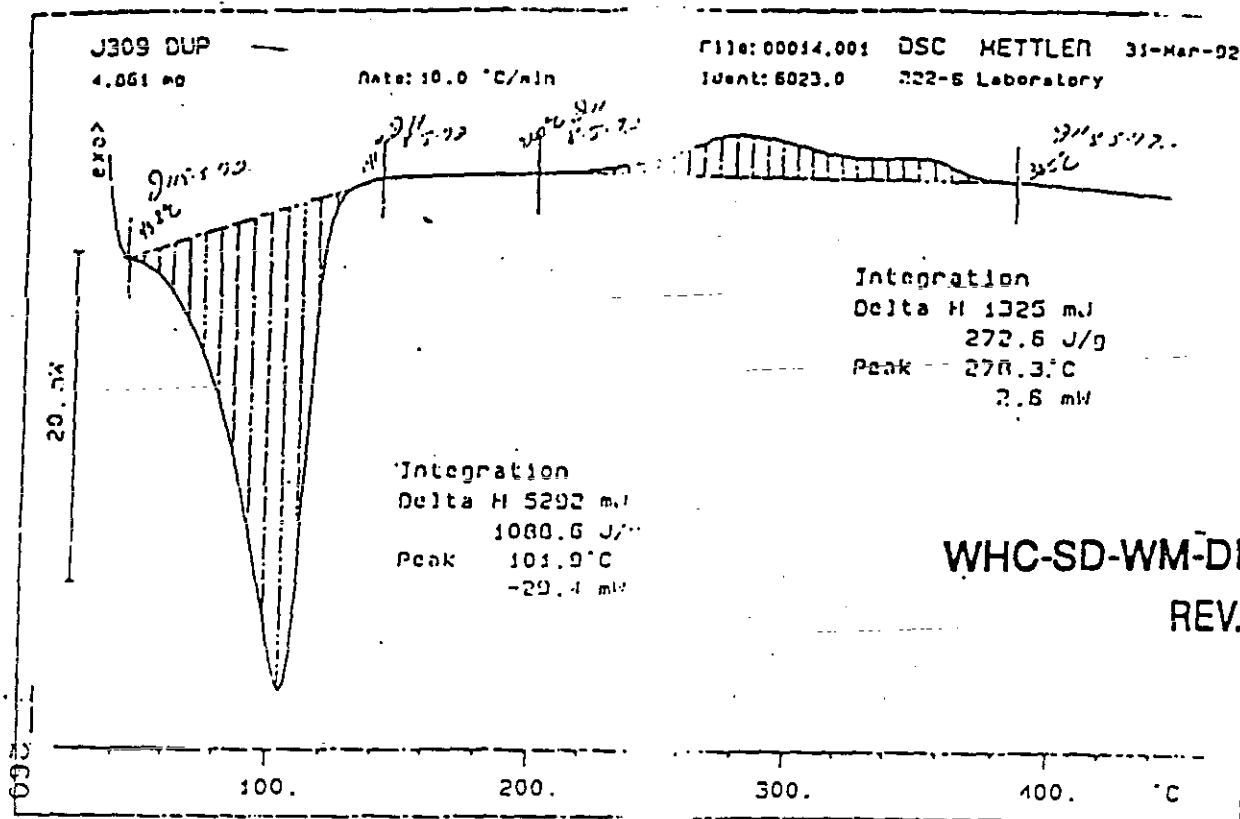
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WHC-SD-WM-DP-058

REV. 0-6

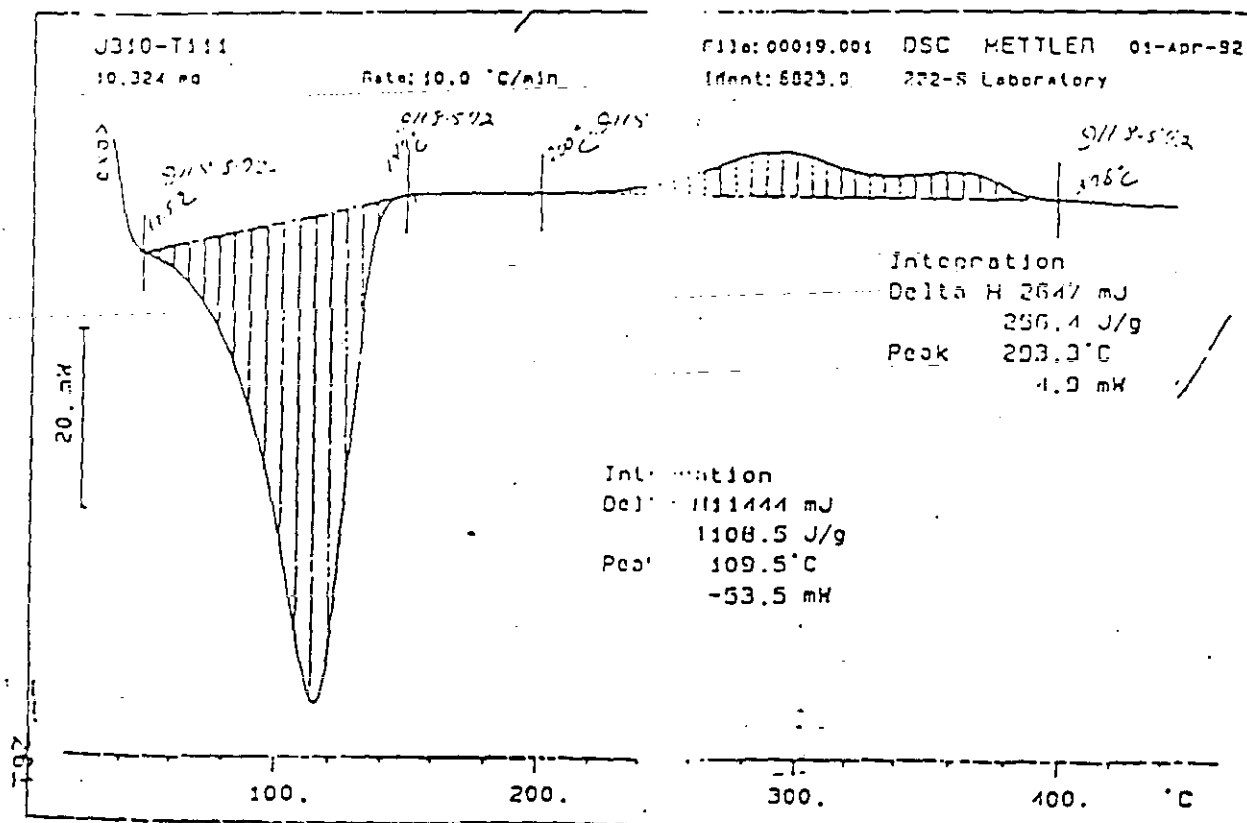
Original T-111 DSC Data 4/92

Core 31 seg 1, wet sample run under static air 4/92



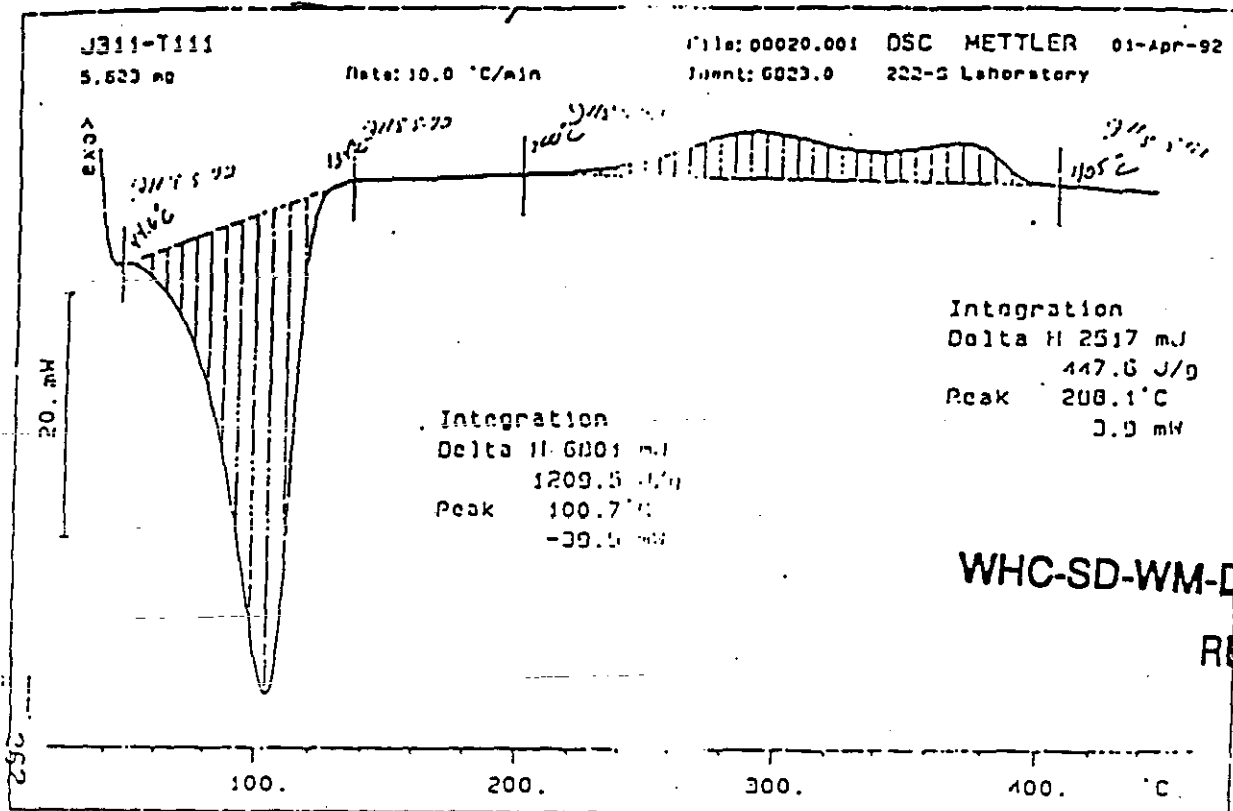
WHC-SD-WM-DP-058, REV 0
Core 31

Core 31 seg 2, wet sample run under static air 4/92

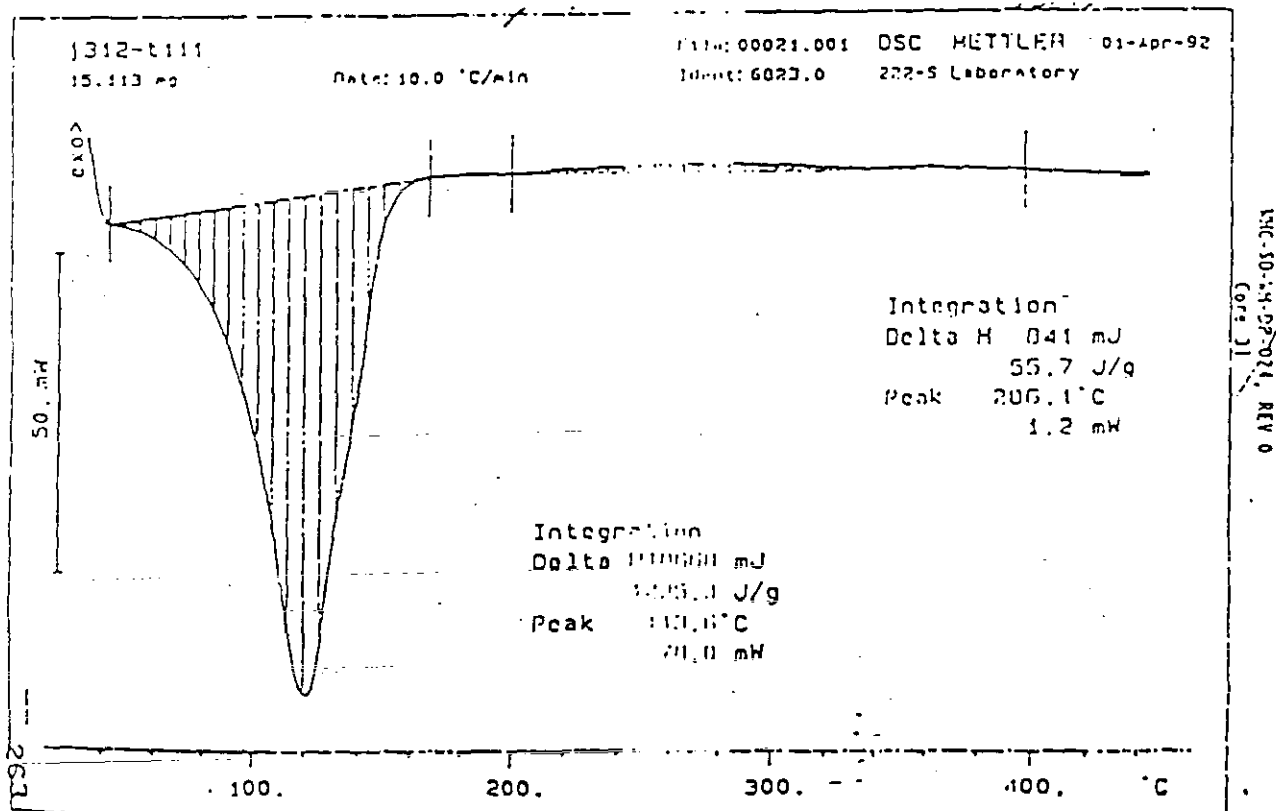


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Core 31

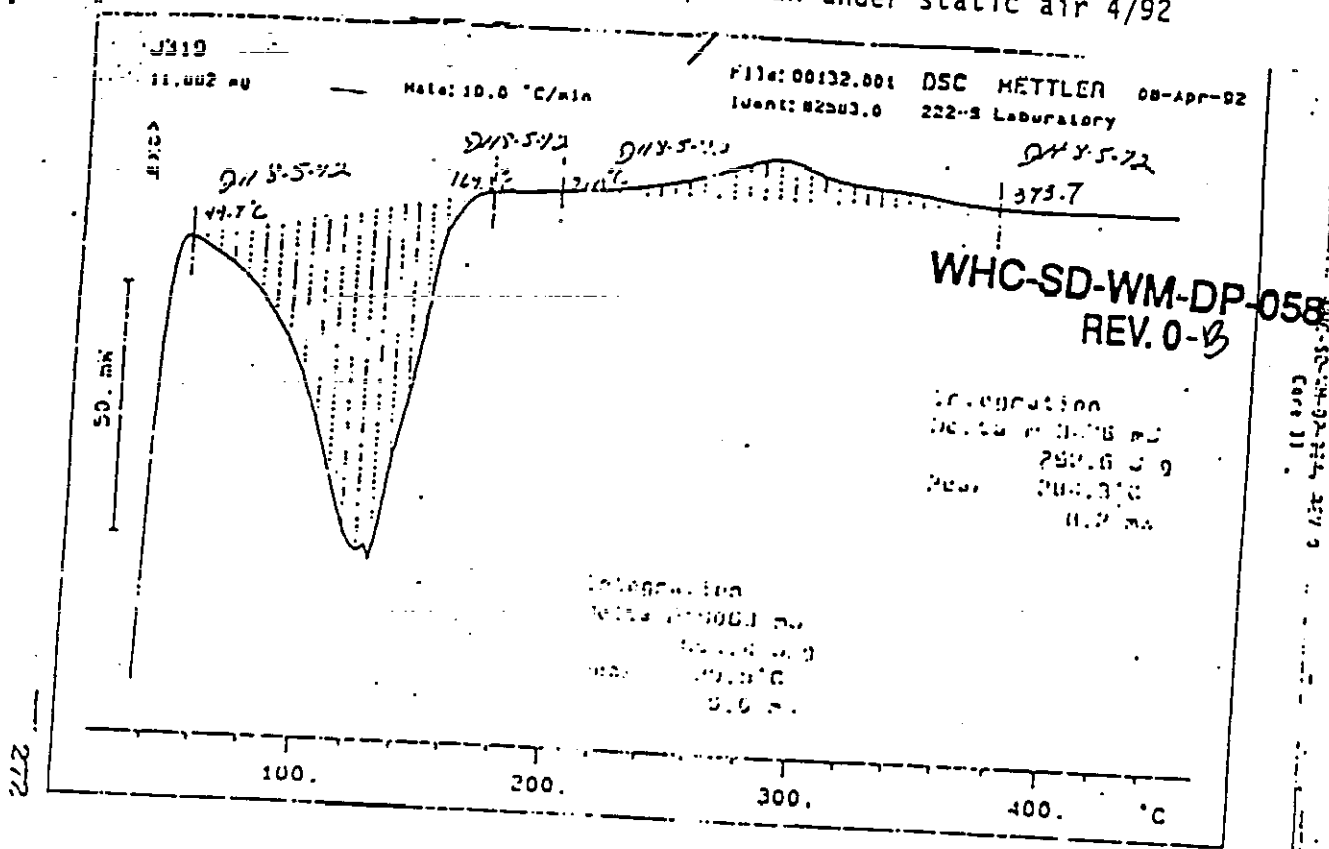
Core 31 seg 3, wet sample run under static air 4/92



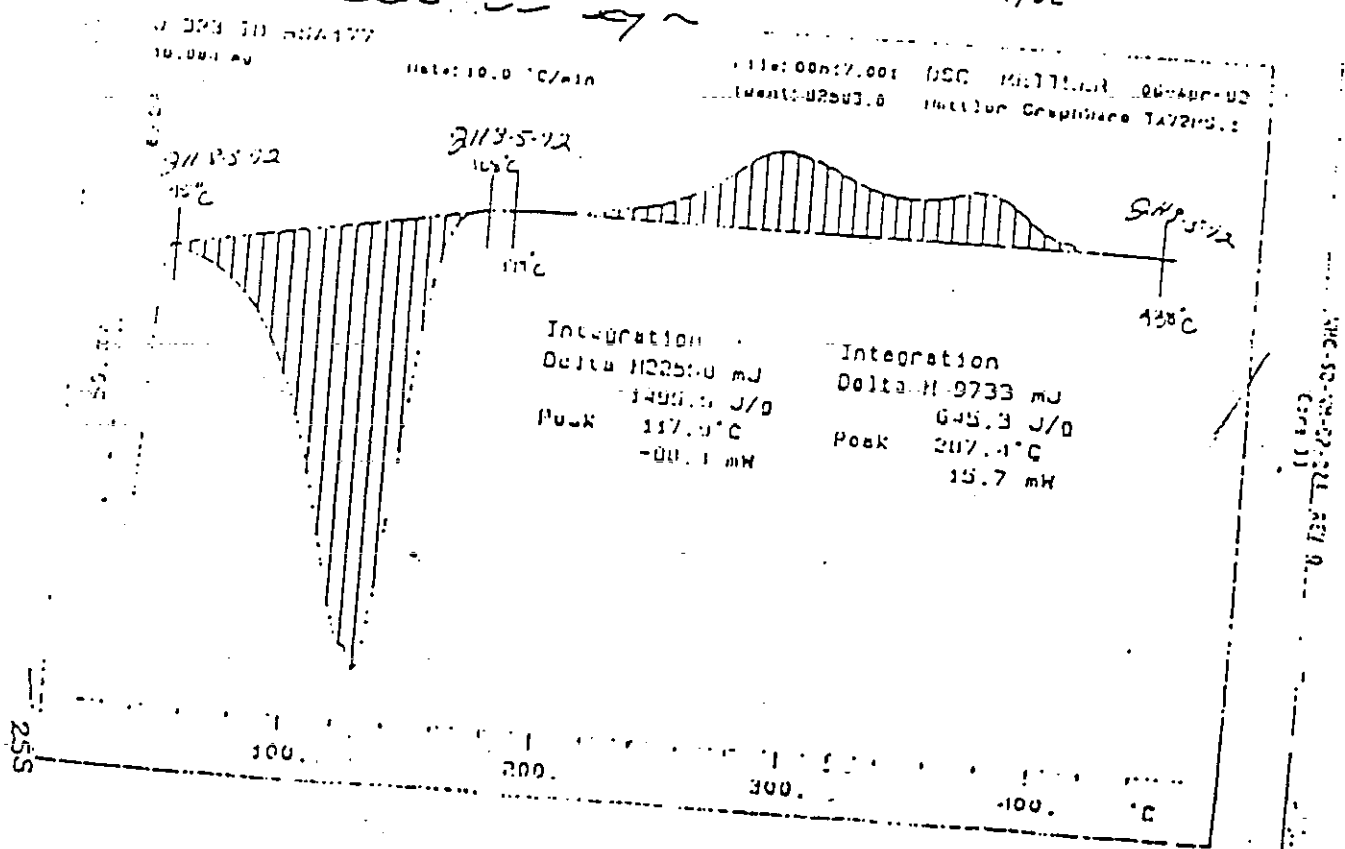
Core 31 seg 4, wet sample run under static air 4/92



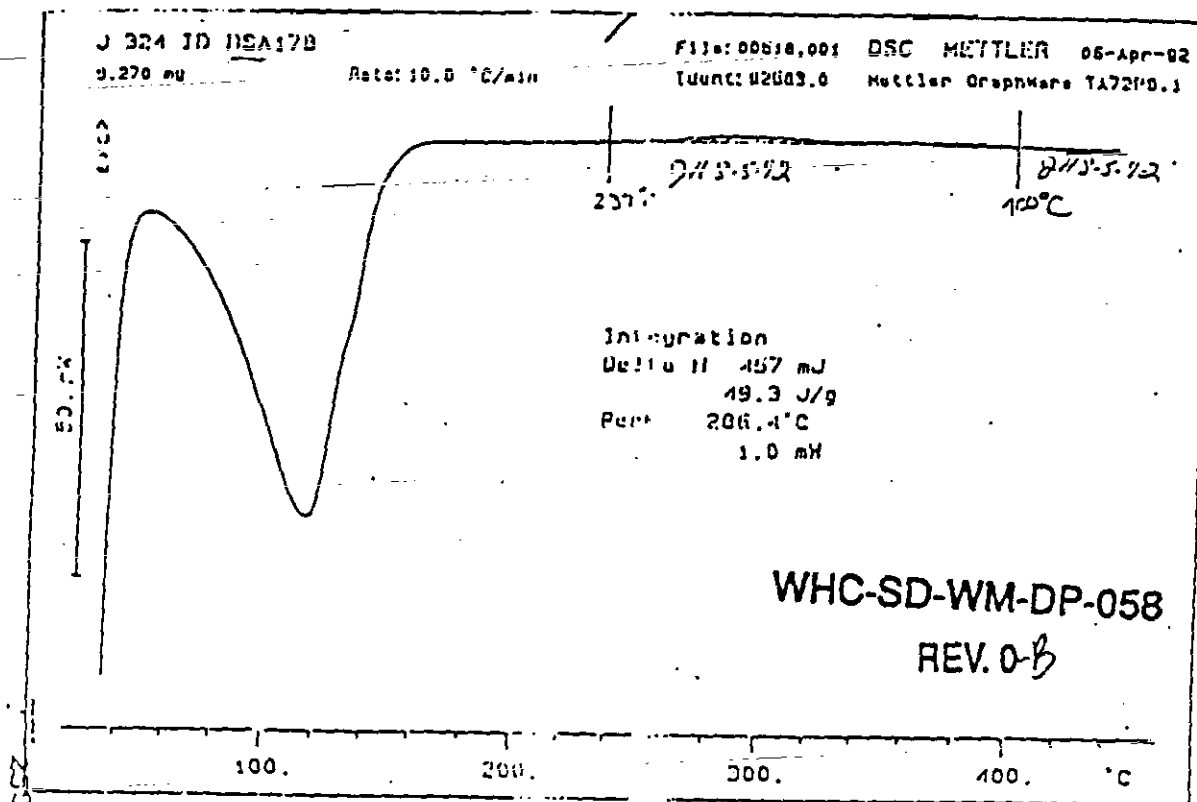
Core 33 seg 1, wet sample run under static air 4/92



Core 33 seg 2, wet sample run under static air 4/92

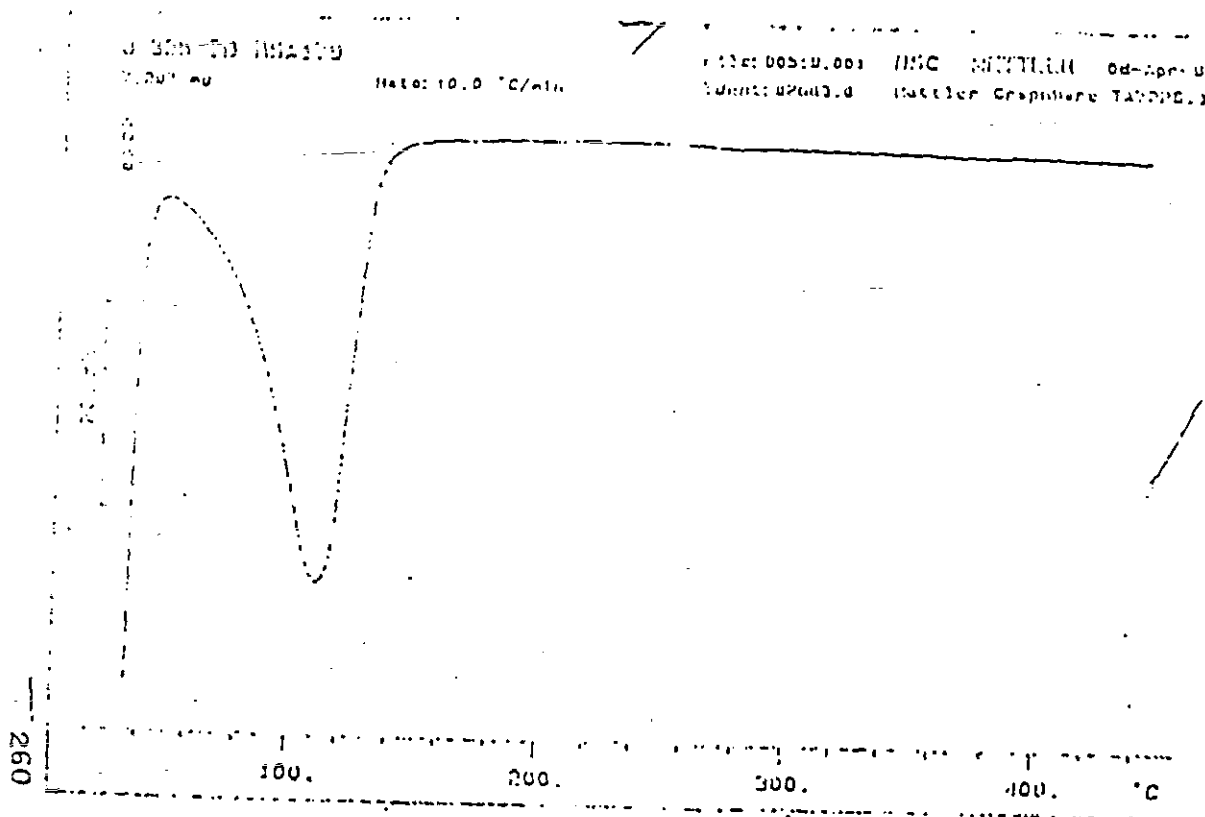


Core 33 seg 3, wet sample run under static air 4/92



WHC-SD-WM-DP-058, REV. 0
Core 33

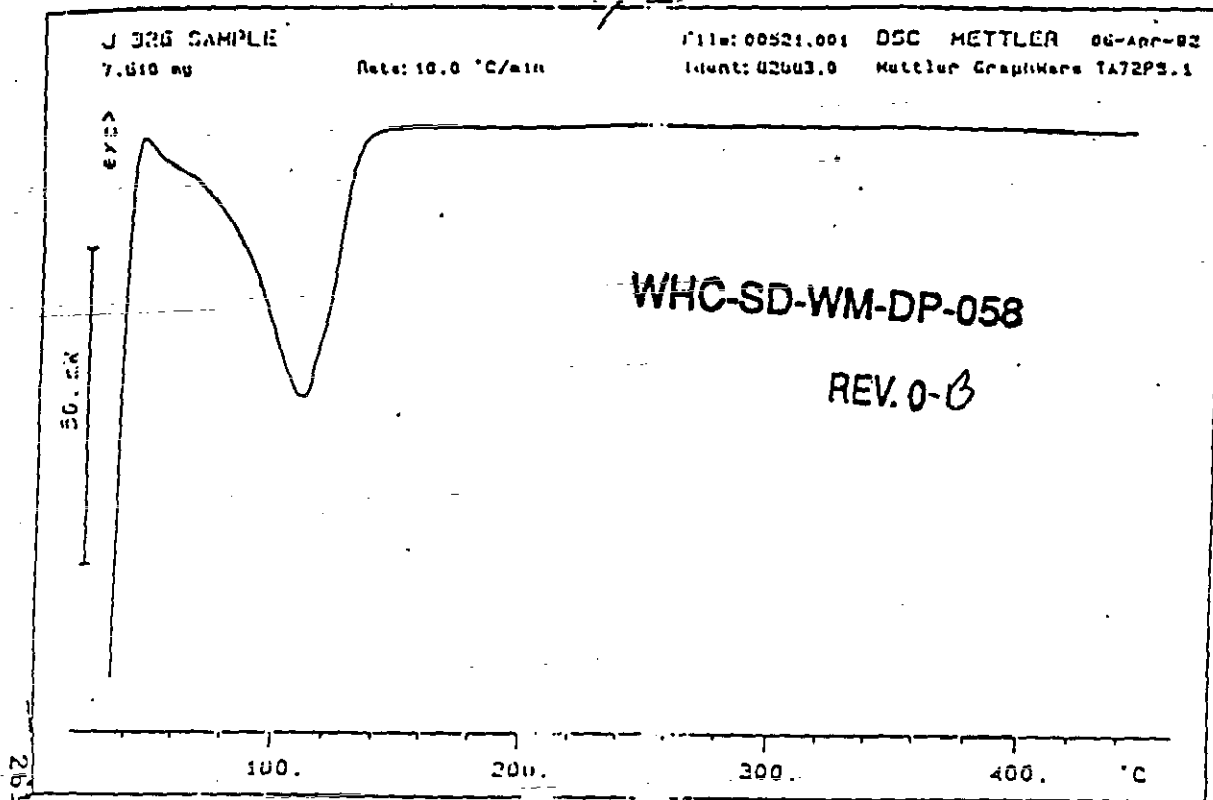
Core 33 seg 4, wet sample run under static air 4/92



WHC-SD-WM-DP-058, REV. 0
Core 33

WHC-EP-0806

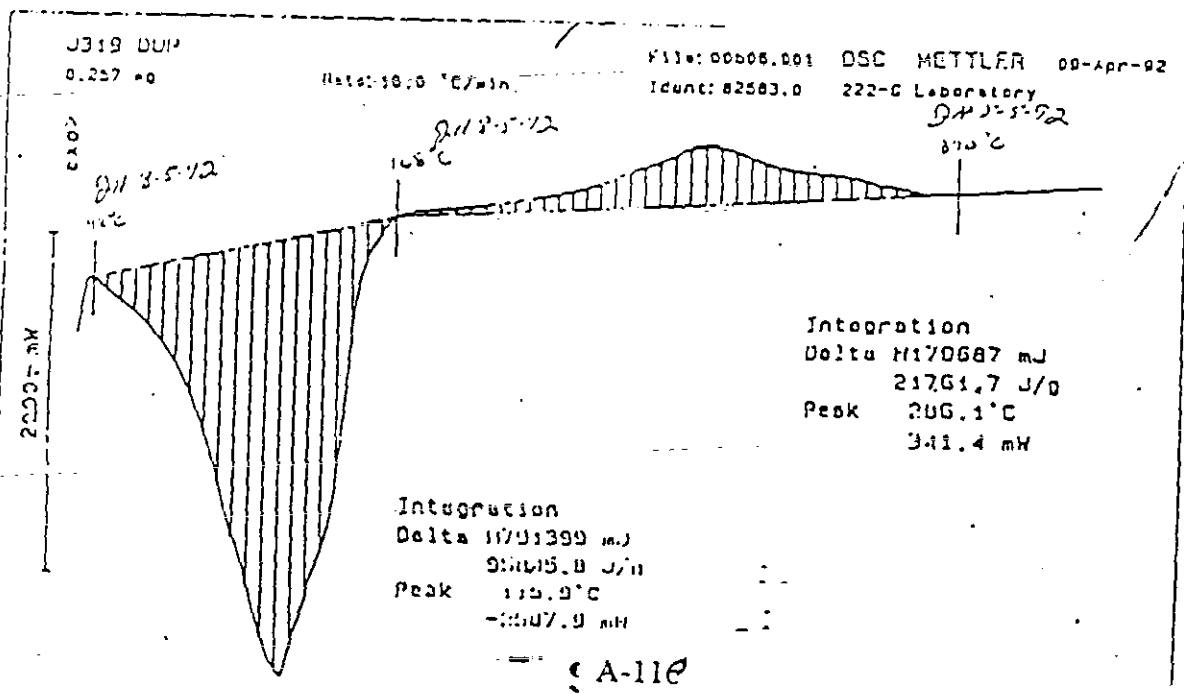
Core 33 seg 5, wet sample run under static air 4/92



2nd. 12.06-92

4/92

Core 31 seg 1, wet sample run under static air 4/92



WHC-EP-0806

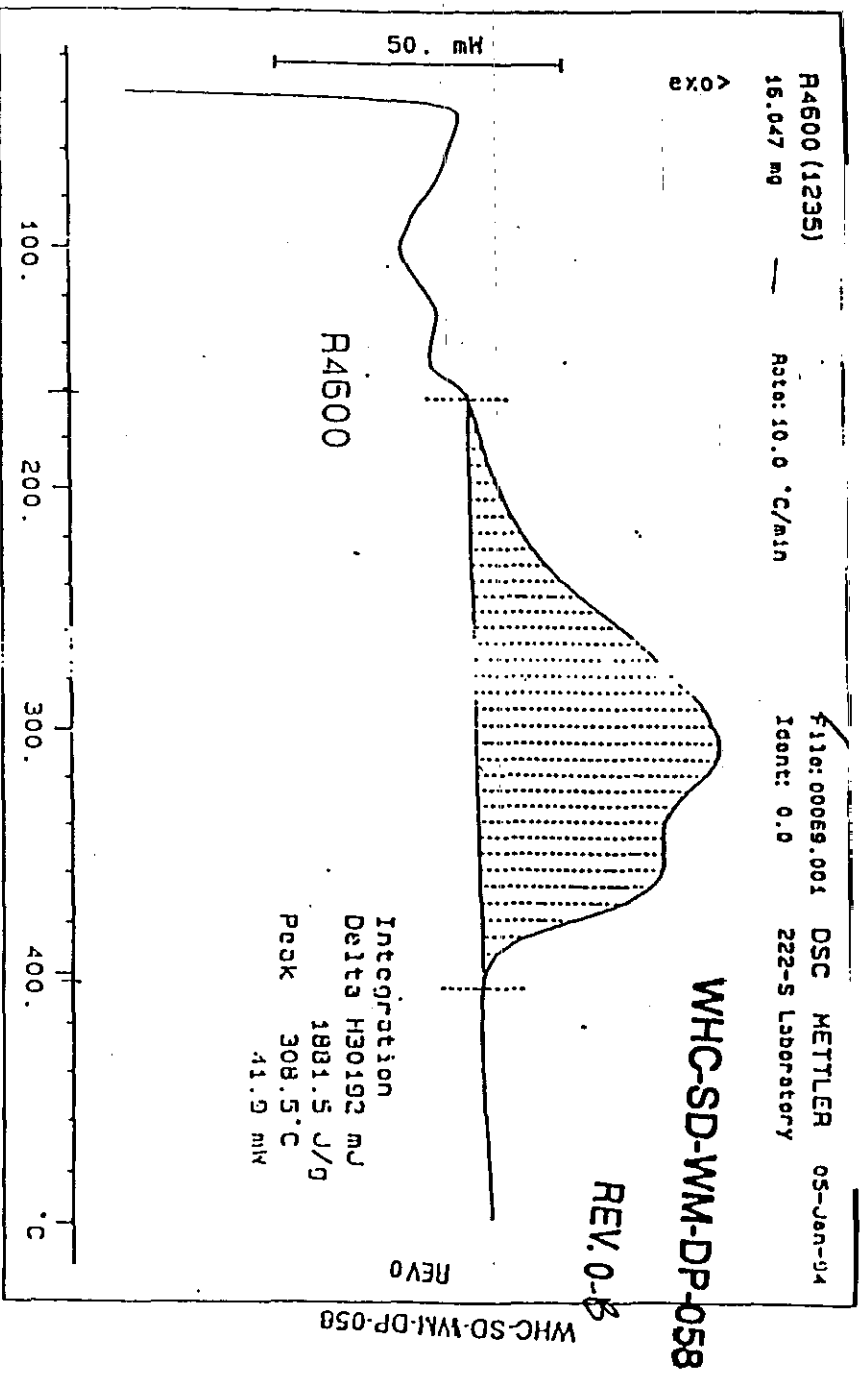
WHC-SD-WM-DP-058

REV. 0-13

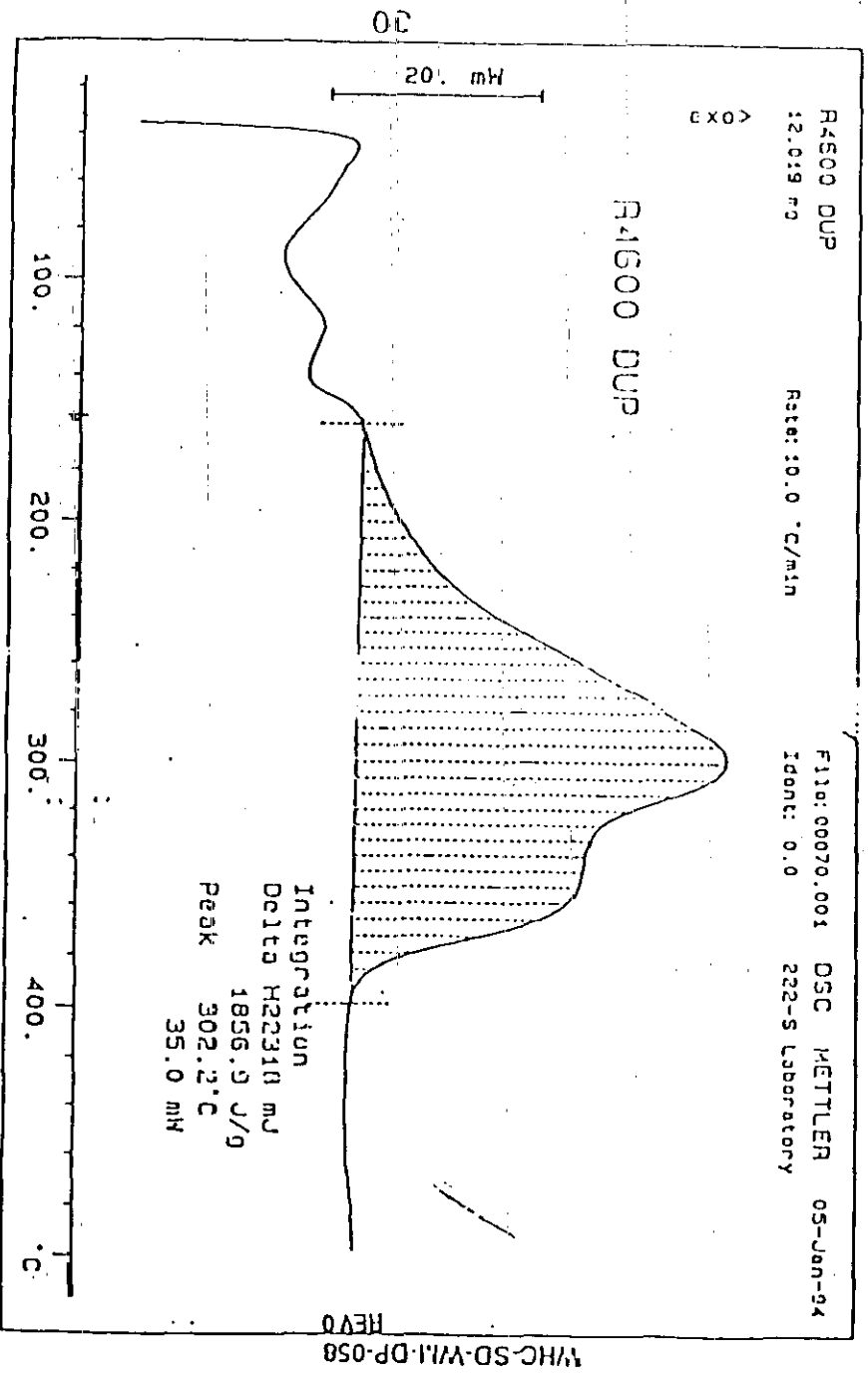
Re-analysis T-111 DSC Data 1/94

WHC-EP-0806

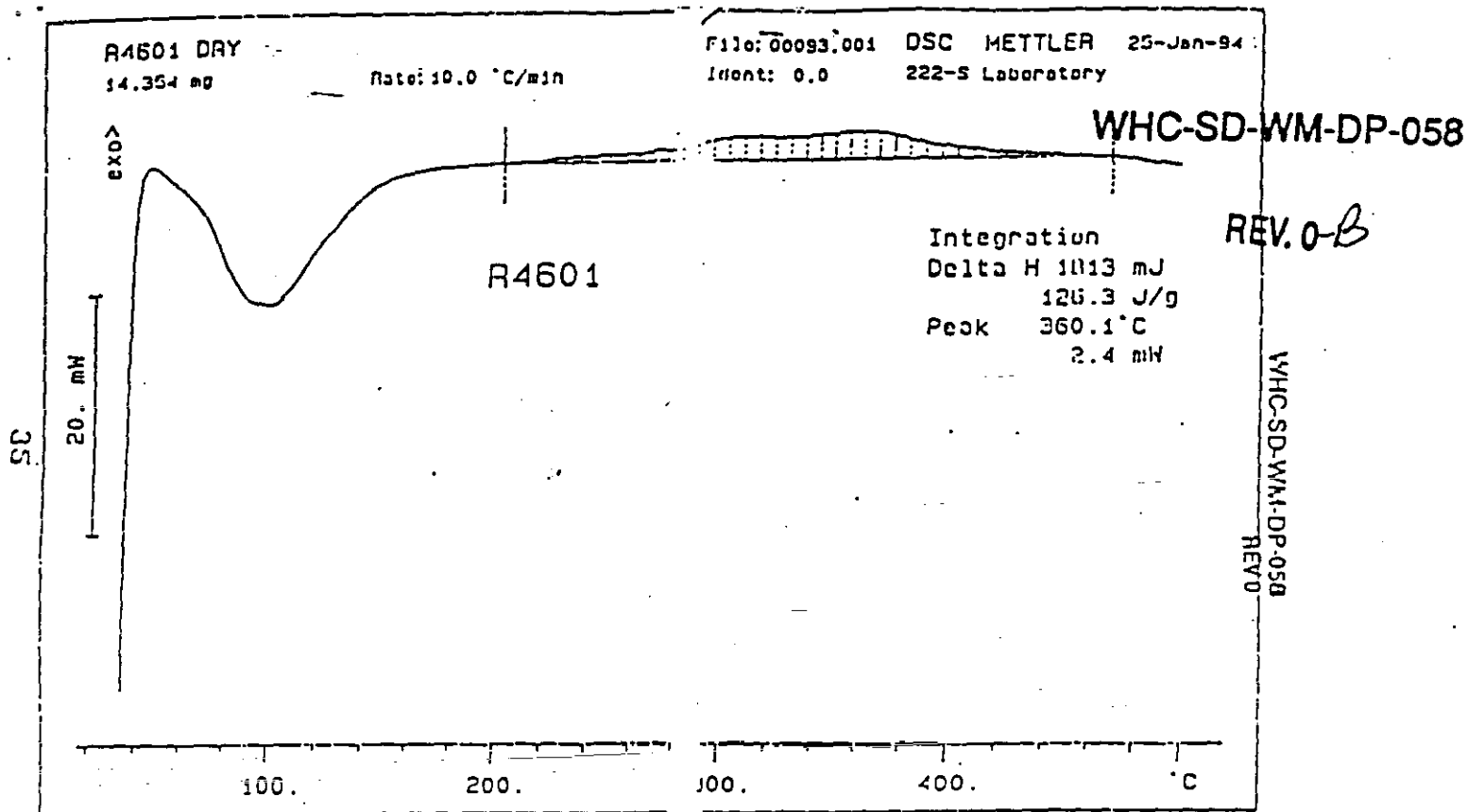
Core 33 seg 1, dried sample run under static air 1/94



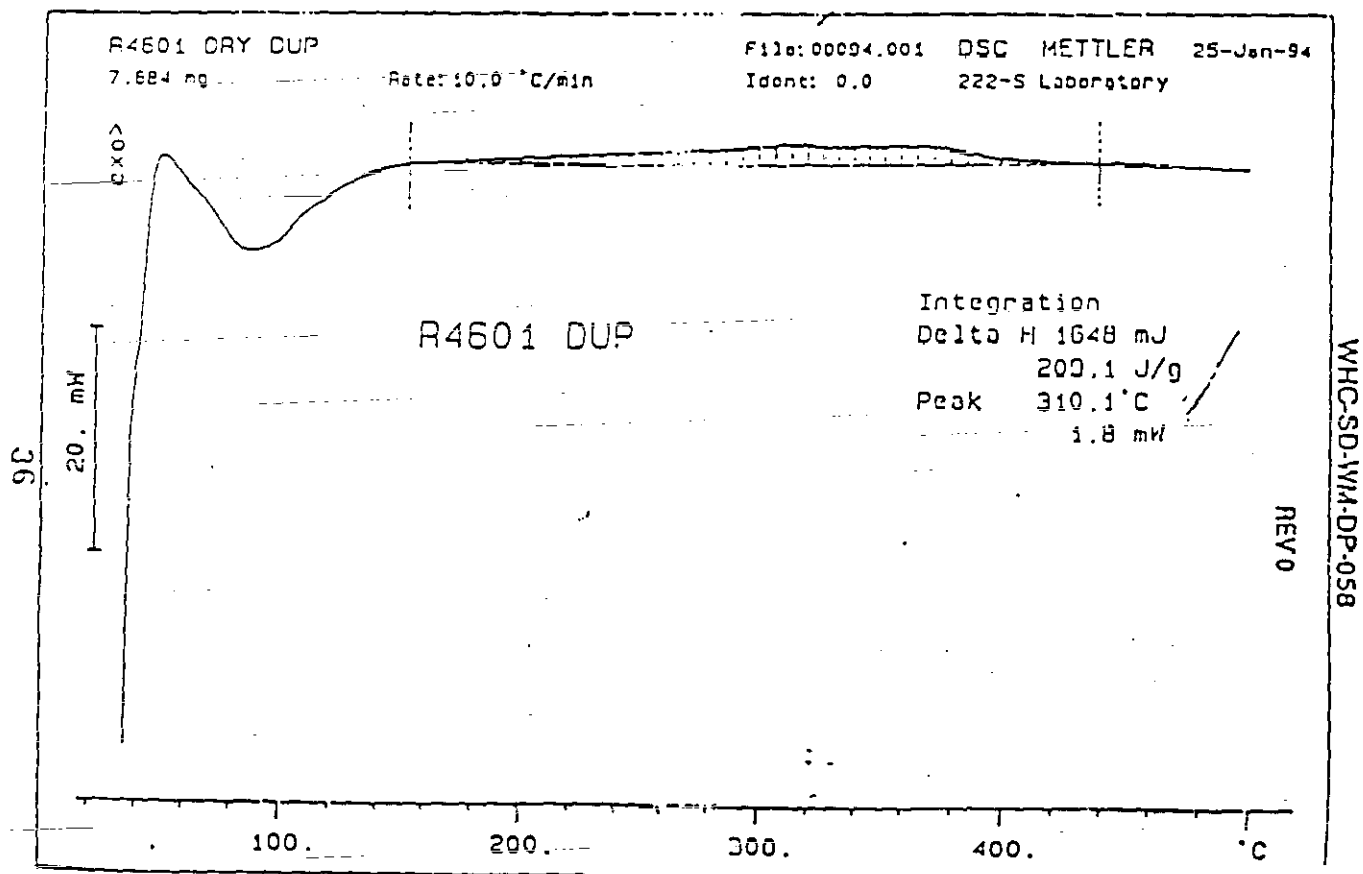
Core 33 seg 1, dried sample run under static air 1/94



Core 33 seg 2, dried sample run under static air 1/94



Core 33 seg 2, dried sample run under static air 1/94

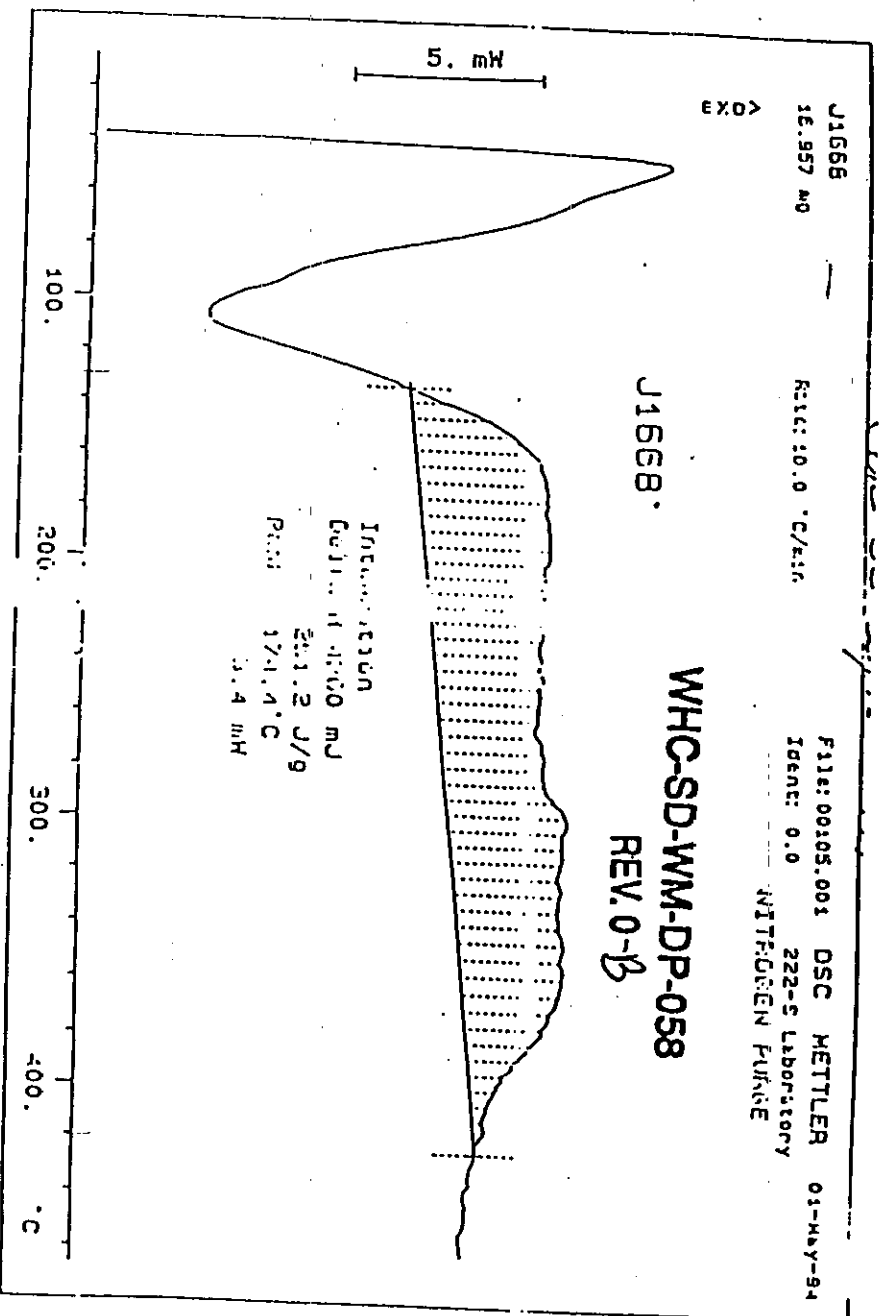


-----WHC-SD-WM-DP-058

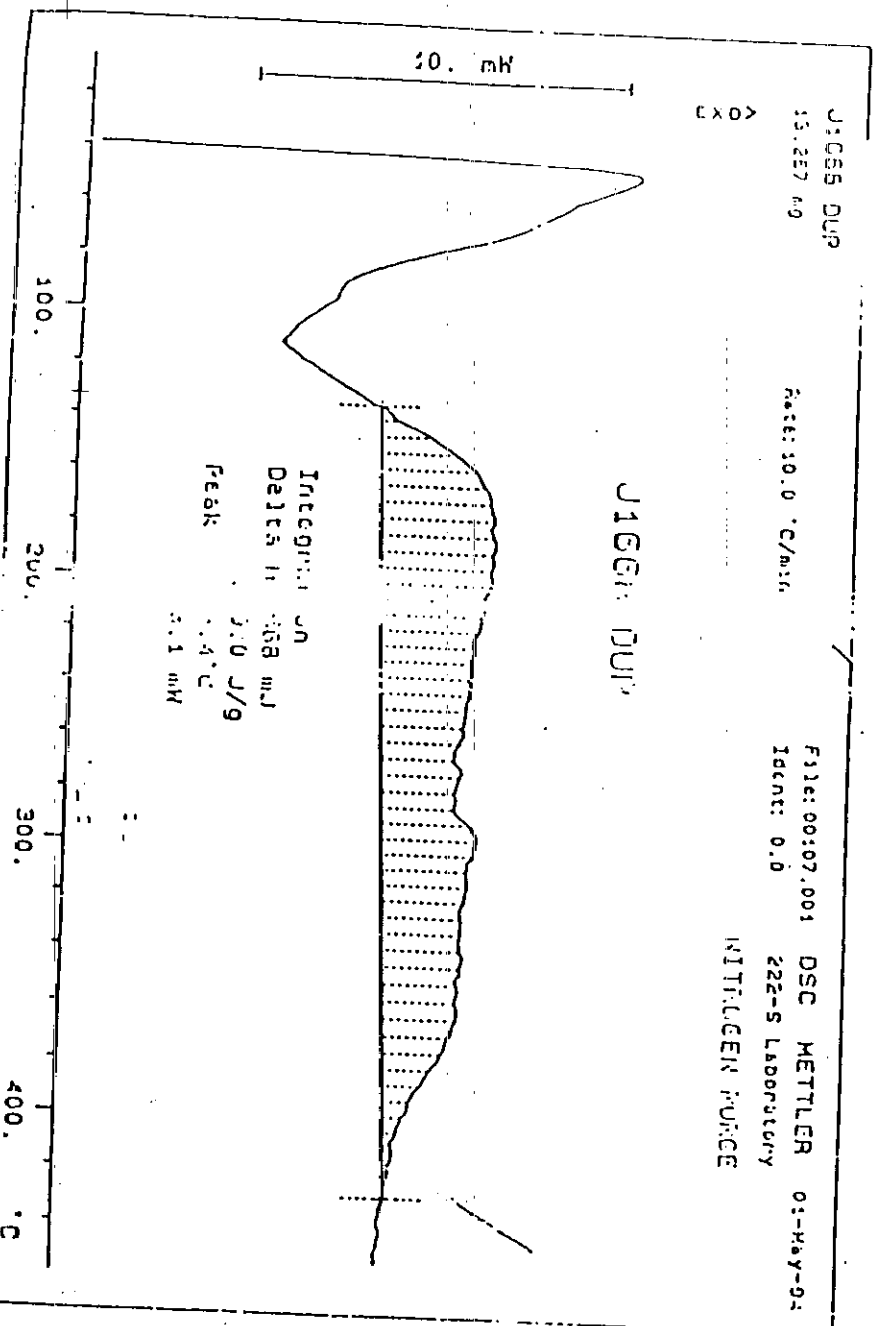
REV. 0- β

-----Re-analysis T-111 DSC Data 5/94

Core 33 seg 2, dried sample run under a nitrogen purge 5/94

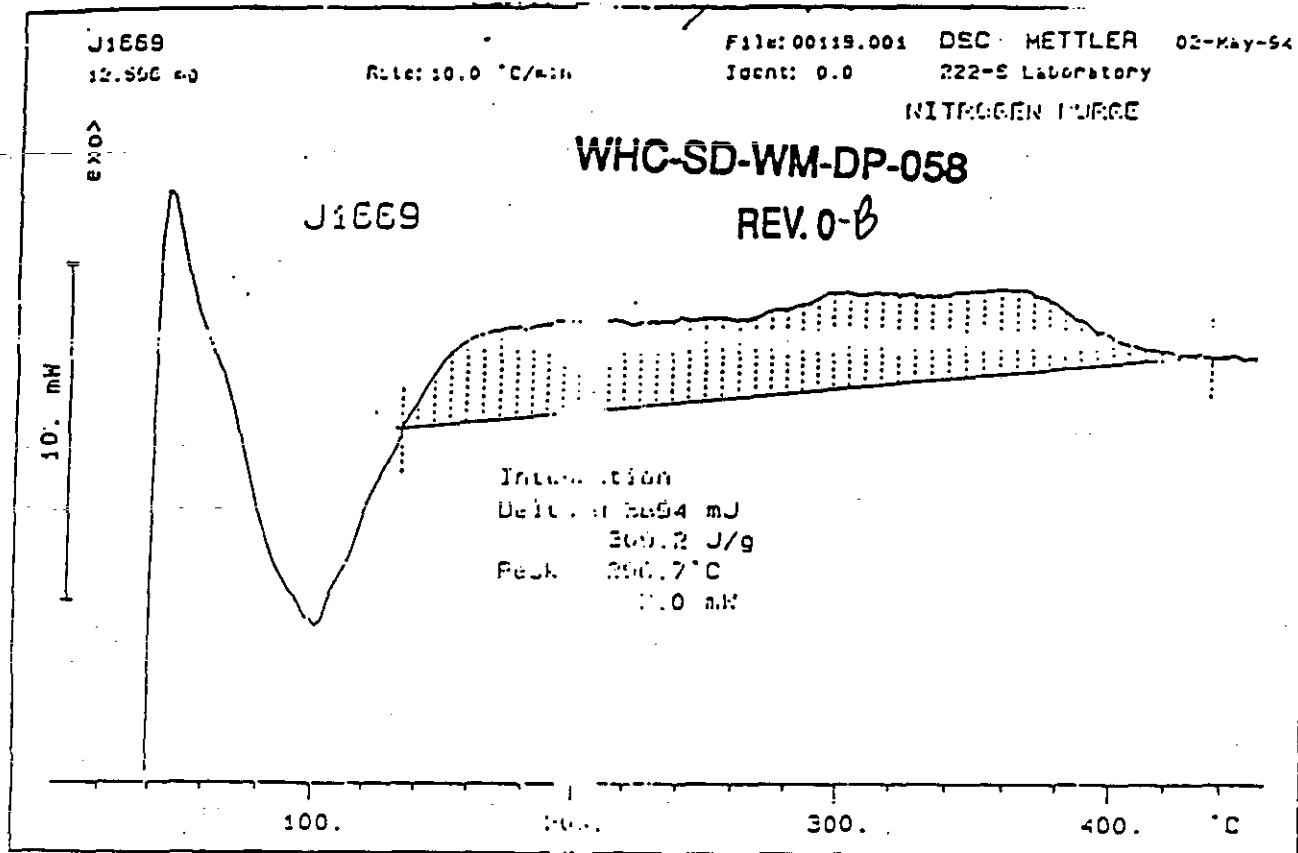


Core 33 seg 2, dried sample run under a nitrogen purge 5/94

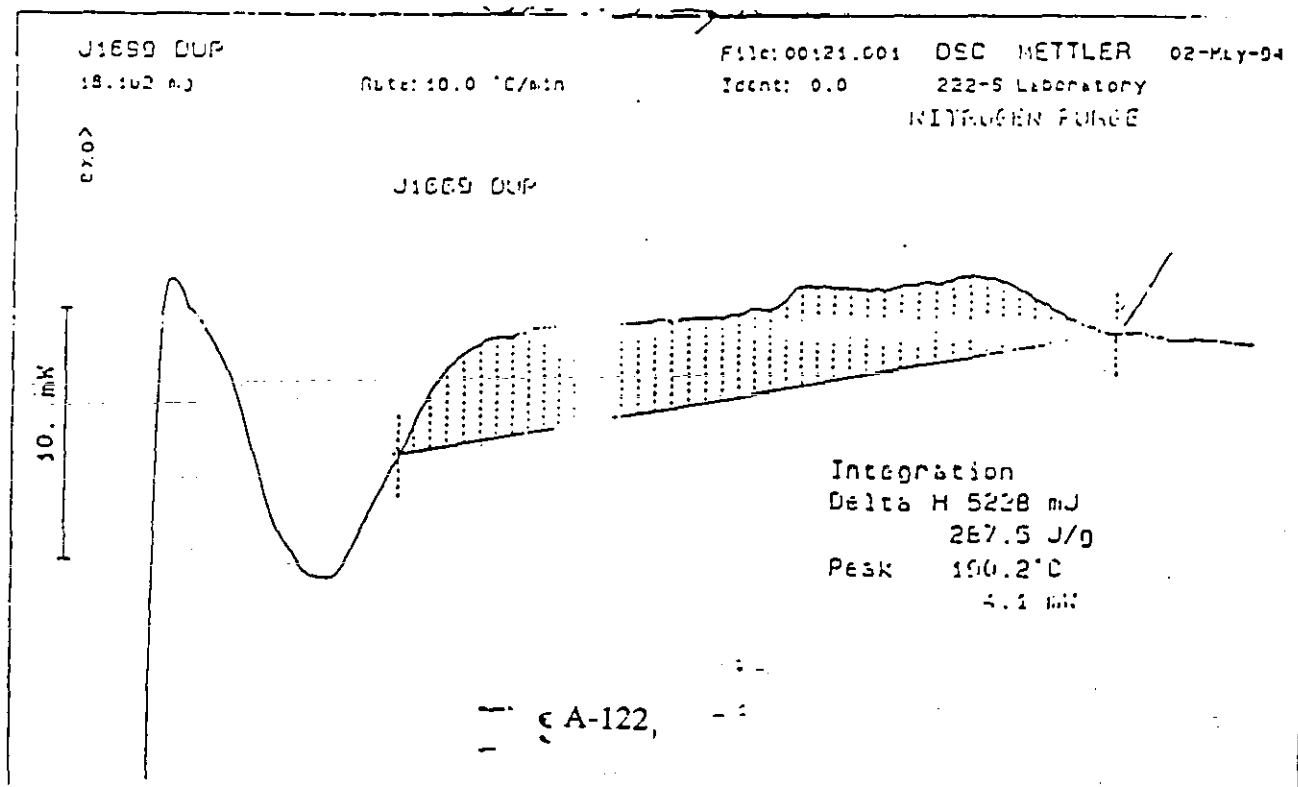


WHC-EP-0806

Core 33 seg 2, dried sample run under a nitrogen purge 5/94

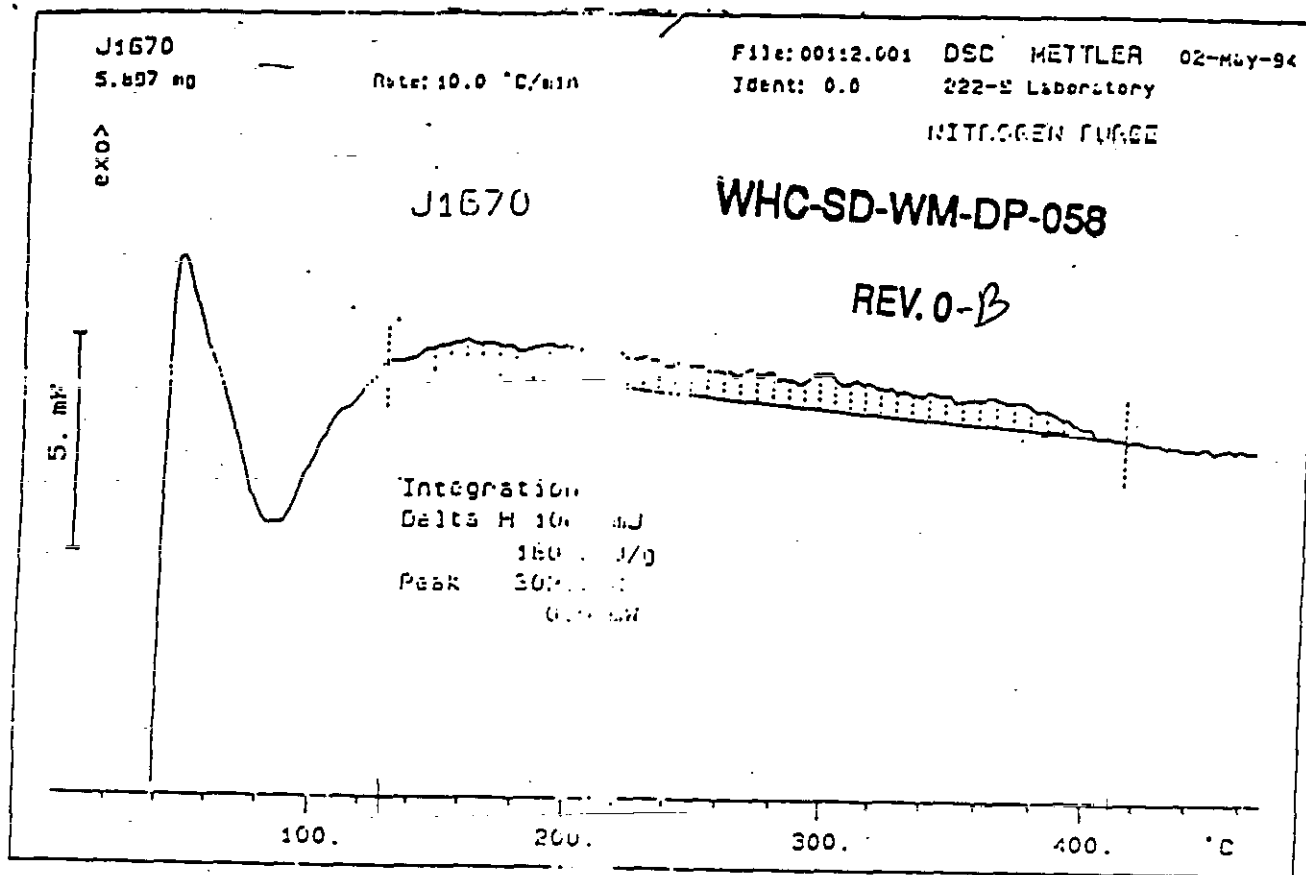


Core 33 seg 2, dried sample run under a nitrogen purge 5/94

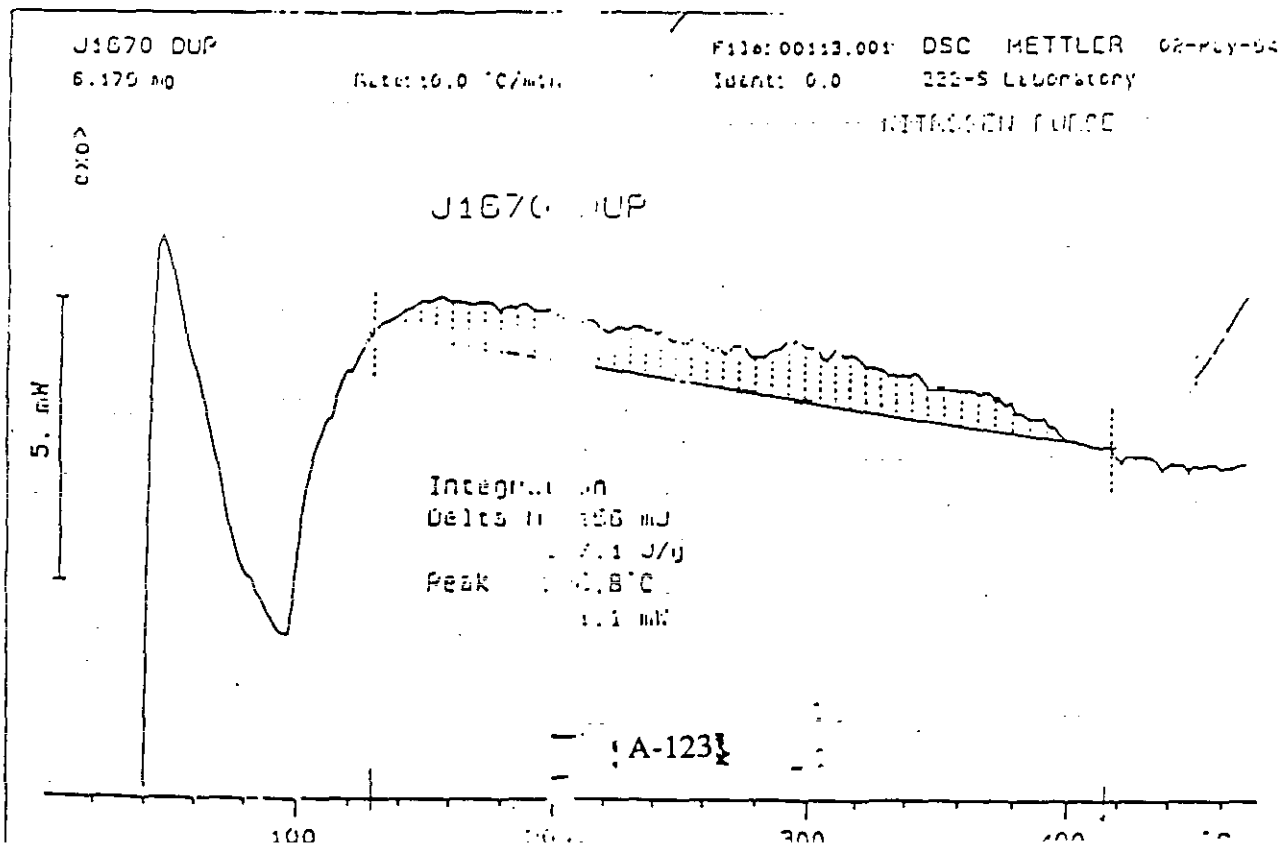


WHC-EP-0806

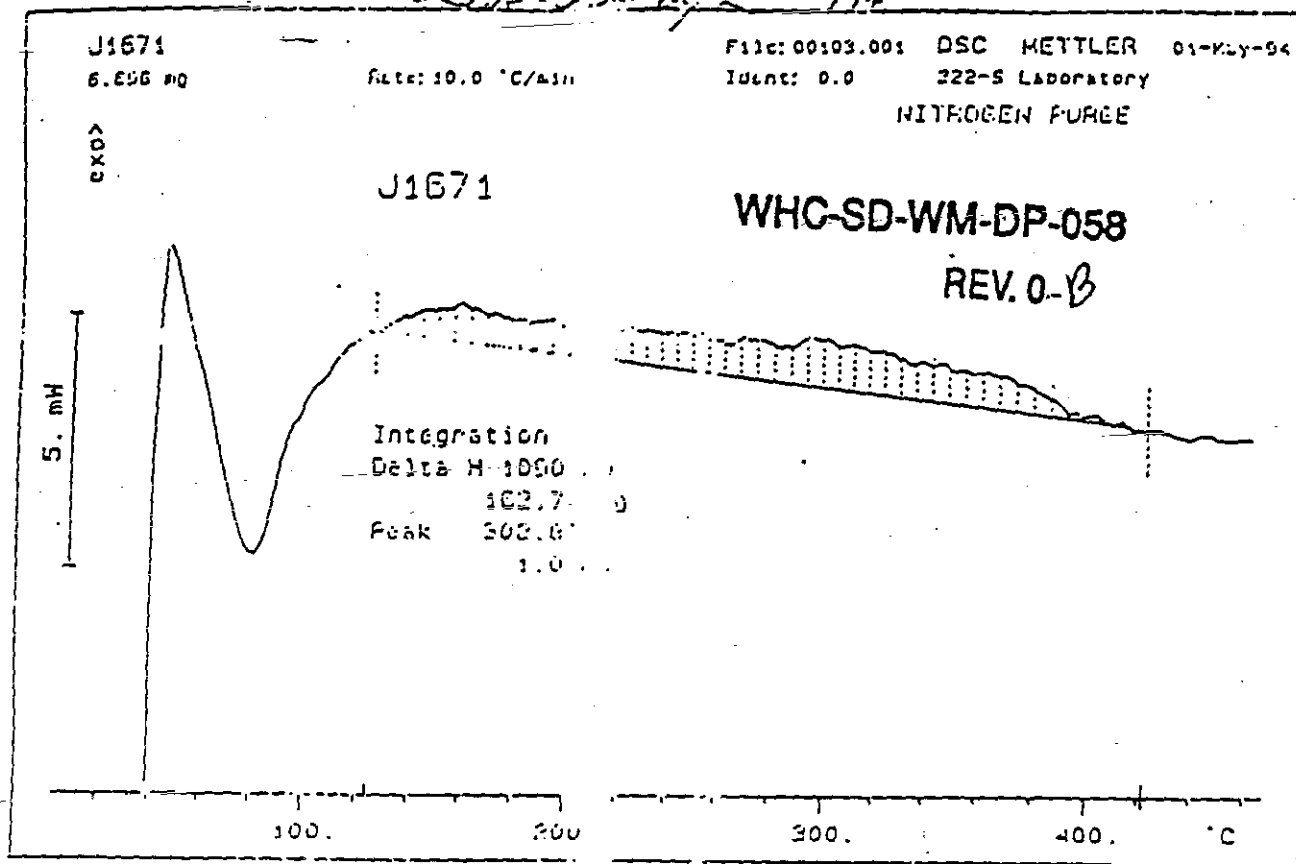
Core 33 seg 2, dried sample run under a nitrogen purge 5/94



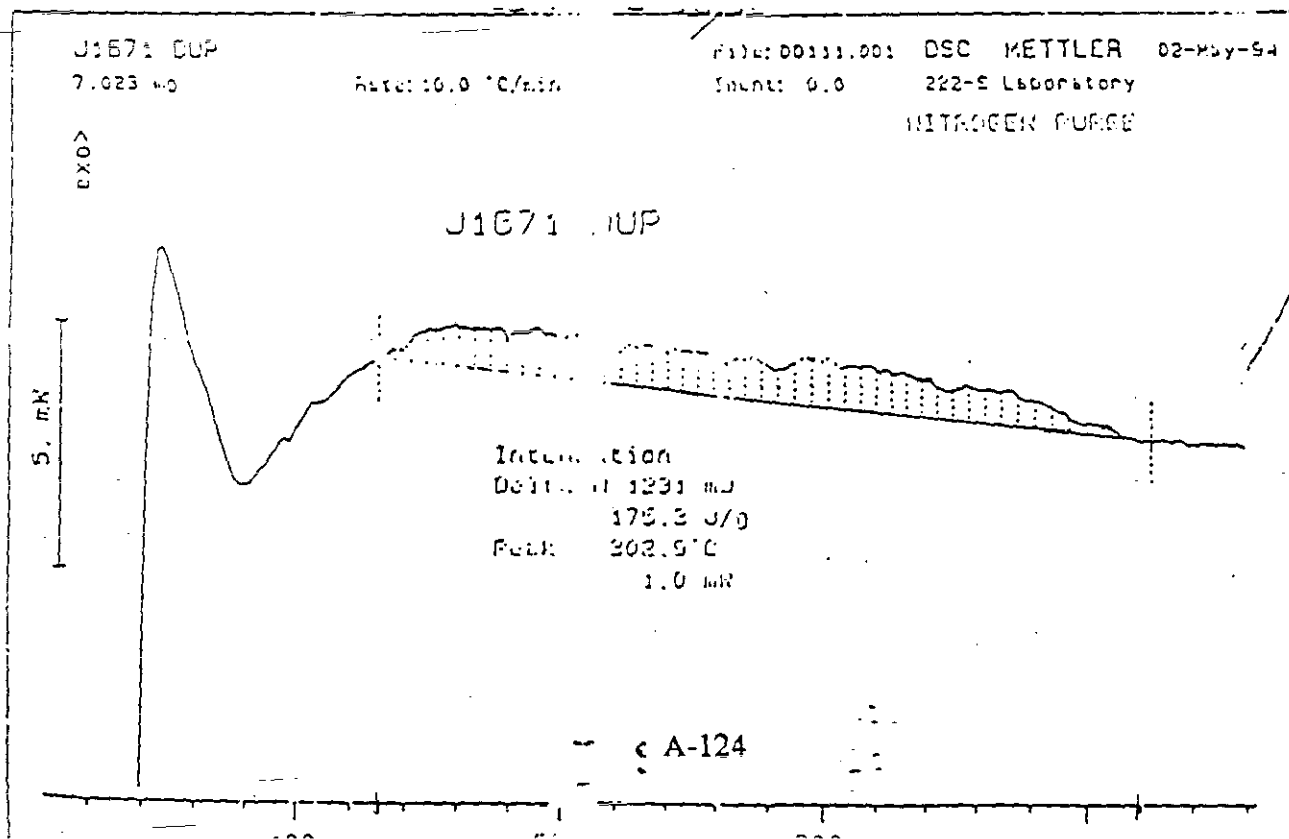
Core 33 seg 2, dried sample run under a nitrogen purge 5/94



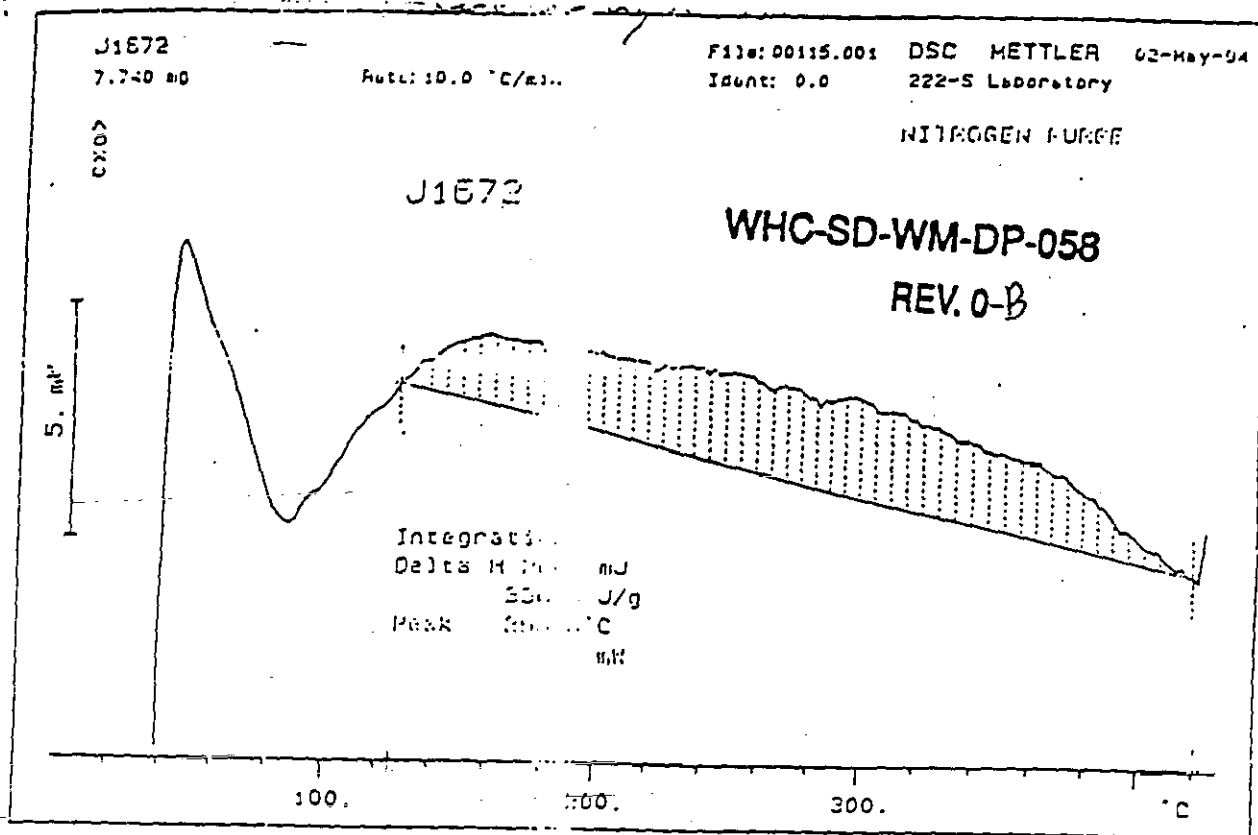
Core 33 seg 2, dried sample run under a nitrogen purge 5/94



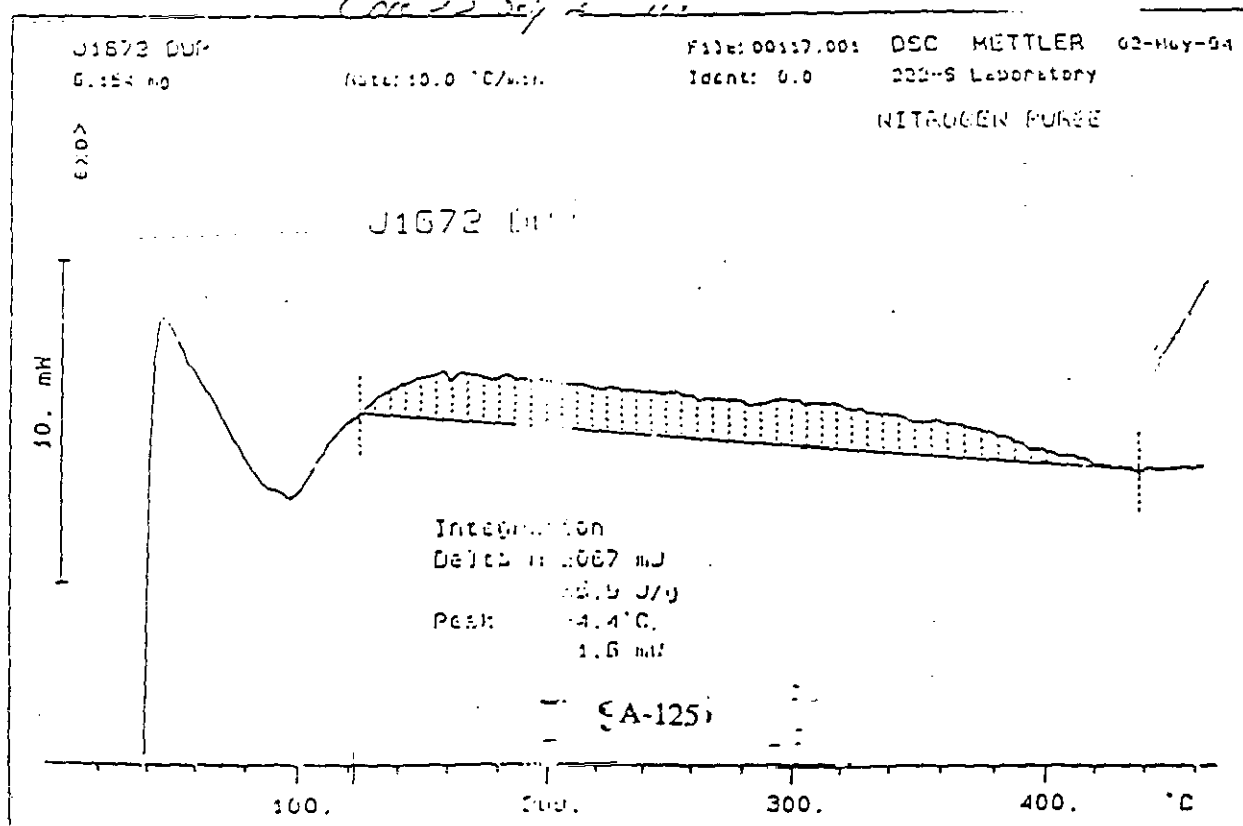
Core 33 seg 2, dried sample run under a nitrogen purge 5/94



Core 33 seg 2, dried sample run under a nitrogen purge 5/94



Core 33 seg 2, dried sample run under a nitrogen purge 5/94



WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-B

PNL Re-Analysis 12/93

WHC-EP-0806

WHC-SD-WM-DP-058

Thermal Analysis Data

REV. 0-3

Thermal analyses of tank waste material from Tank 241-T-111 Core 33, Segment 2 were performed in duplicate. The thermal analysis includes both differential scanning calorimetry (DSC) and scanning thermogravimetry (TGA). DSC analyses were performed on dried sample material, but the TGA analyses were performed on the as-received sample. These analyses were performed according to technical procedure PNL-AL0-508, "Laboratory Procedure for Operation of the Differential Scanning Calorimeter (DSC), Thermogravimetric Analyzer (TG), and High Temperature Differential Thermal Analyzer (DTA) and DSC."

dried { DSC analyses of sample 92-05856-N and it's duplicate were performed on a Perkin Elmer Model 2 Differential Scanning Calorimeter. The sample was prepared by transferring 0.4896 grams of sample from 92-05856 (labeled T-111, Core 33, Segment 2) to a glass vial. This sample was placed in a vacuum oven at 60°C and 28 in. Hg (711 mm Hg) at 1315 hours on December 9, 1993. The sample was removed from the oven at 1300 hours on December 10, 1993 and allowed to cool prior to weighing. The sample weight after this drying period was 0.0721 grams (a loss of 0.4175 grams or 85.3% mass loss). The sample was placed back in the oven at 60°C and 28 in. Hg at 1310 hours on December 10, 1993 and removed at 1500 hours on the same day. The sample was reweighed and a loss of only 0.0005 grams was observed; therefore, the sample was sealed and placed in a desiccator. The sample was analyzed on December 13, 1993 using a nitrogen gas environment. Samples of 11.19 and 15.72 milligrams were used for Runs 1 and 2, respectively. A temperature range from 35 to 550°C was scanned at 5°C/min. The results of these analyses are given in Table 1-1.

Table 1-1: Core 33, Segment 2, Energetics

RUN	RANGE (°C)	ONSET (°C)	ENTHALPY (J/g)
1	107 - 381	201	-898
2	113 - 394	196	-836

WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-B

An indium and a zinc standard was run prior to analyzing the sample. The indium standard had an onset temperature of 156.8°C with an enthalpy of 28.3 J/g. The literature values for indium are 156.6°C and 28.45 J/g. The zinc standard had an onset temperature of 417.1°C with an enthalpy of 98.7 J/g. The literature values for zinc are 419.47°C and 108.37 J/g.

Thermogravimetric analysis was performed in triplicate on an aliquot of as-received sample material using a Perkin Elmer Model 2 Thermogravimetric Analyzer. A nitrogen gas environment was used to analyze these samples. Samples of 11.32, 17.11, and 19.40 milligrams were used for Runs 1, 2, and 3, respectively. Run 1 was performed on December 28, Run 2 on December 29, 1993, and Run 3 on January 3, 1994. A temperature range from 35 to 550°C was scanned at 5°C/min for Runs 1 and 2. The temperature range for Run 3 was from 35 to 200°C at a scan rate of 5°C/min. This shorter scan range was chosen, because no significant weight loss was observed above 150°C. The results of these analyses are given in Table 1-2.

Table 1-2: Core 33, Segment 2, Mass Loss as a Function of Temperature

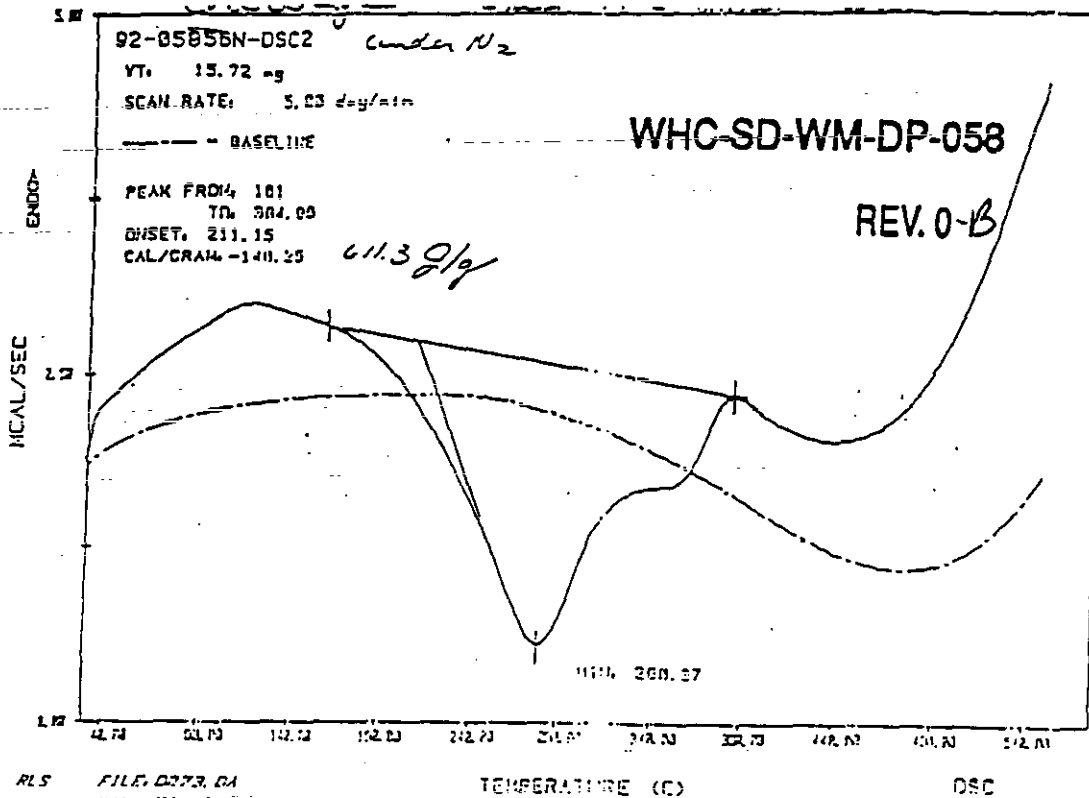
RUN	RANGE (°C)	WEIGHT LOSS (wt%)
1	31 - 103	67.3
2	31 - 116	80.3
3	30 - 130	79.7

Run 1 appeared to be on a sample that was significantly drier than the other samples. Runs 2 and 3 compare well with the gravimetric/determinations weight percent solids measurements. It was also noted in Runs 1 and 2 that no significant weight loss was observed in the temperature range of the exothermic reaction.

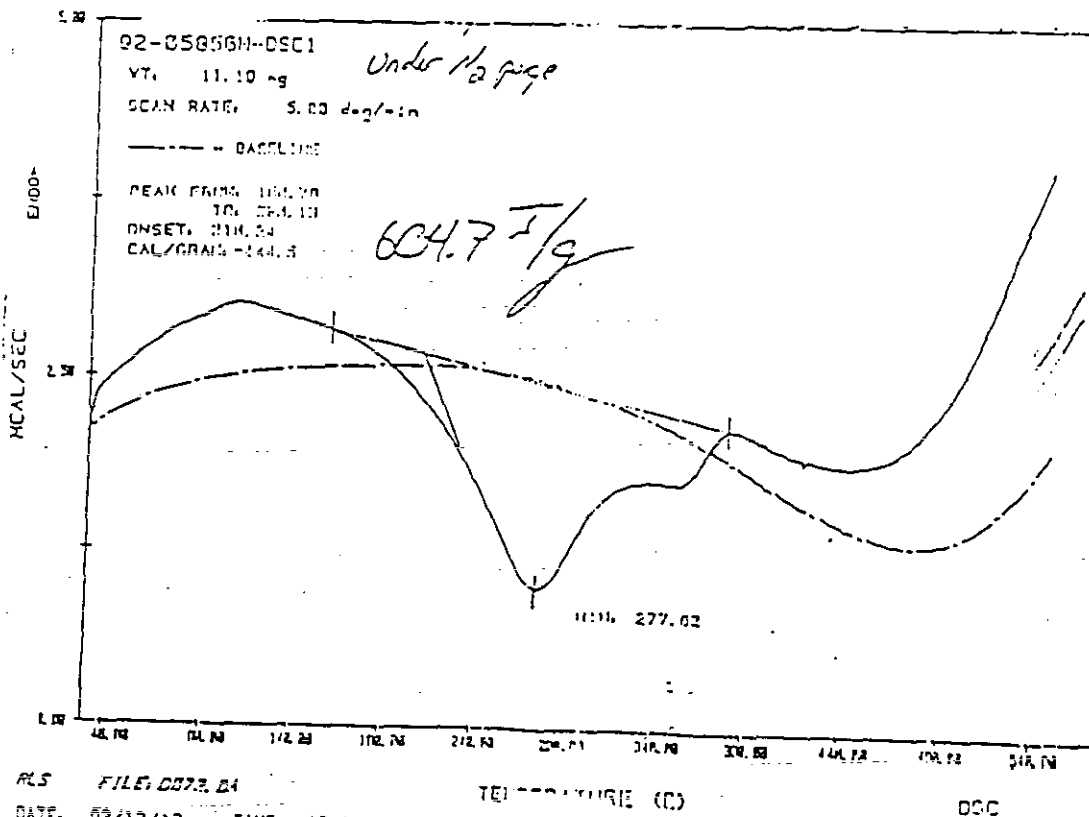
Alumel and perkallloy curie point standards were run prior to analyzing the sample. The alumel standard had an onset temperature of 162.35°C, and the perkallloy standard had an onset temperature of 595.73°C. The literature values for alumel and perkallloy are 163°C and 596°C, respectively.

WHC-EP-0806

Core 33 seg 2, dried sample run under a nitrogen purge 12/93



Core 33 seg 2, dried sample run under a nitrogen purge 12/93



WHC-SD-WM-DP-058

Rev. 0-13

Jan 3/24/94

@@:PPCALIBRATION 2

17-FEB-94 8:45

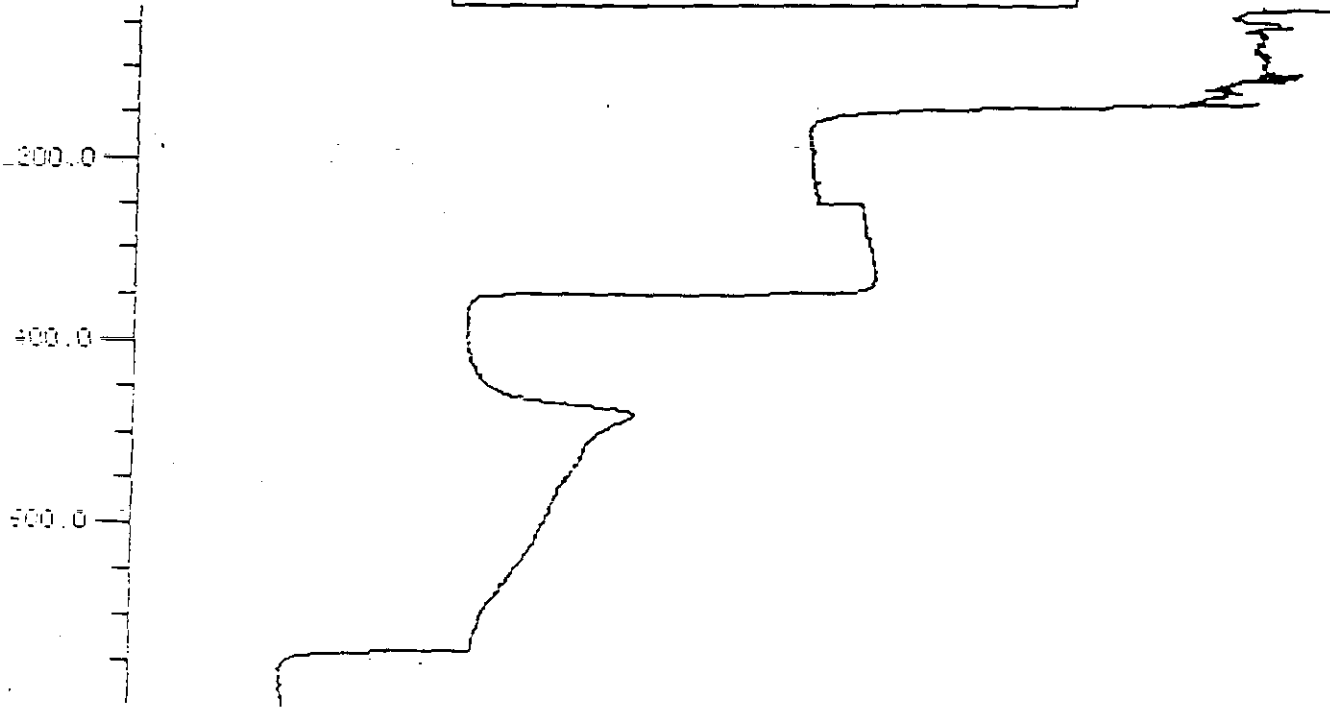
START TEMP. °C 35
END TEMP. °C 800

FILE NO. 00130.001
IDENT. NO. 0
WEIGHT mG 6.7520

TEMPERATURE °C

WEIGHT
GAIN----->

5.0000 mG



TEMP. °C 143.67
TEMP. °C 349.67
TEMP. °C 739.00

James L. Juge
2/17/94

A PT100 -22341
B PT100 -70428
C PT100 -05962

A-130

James L. Juge
2/17/94

WHC-EP-0806

CALIBRATION 2

31-JAN-94 16:16

START TEMP. °C 35
END TEMP. °C 750.

FILE NO. 00120.001
IDENT. NO. 0
WEIGHT mg 7.57

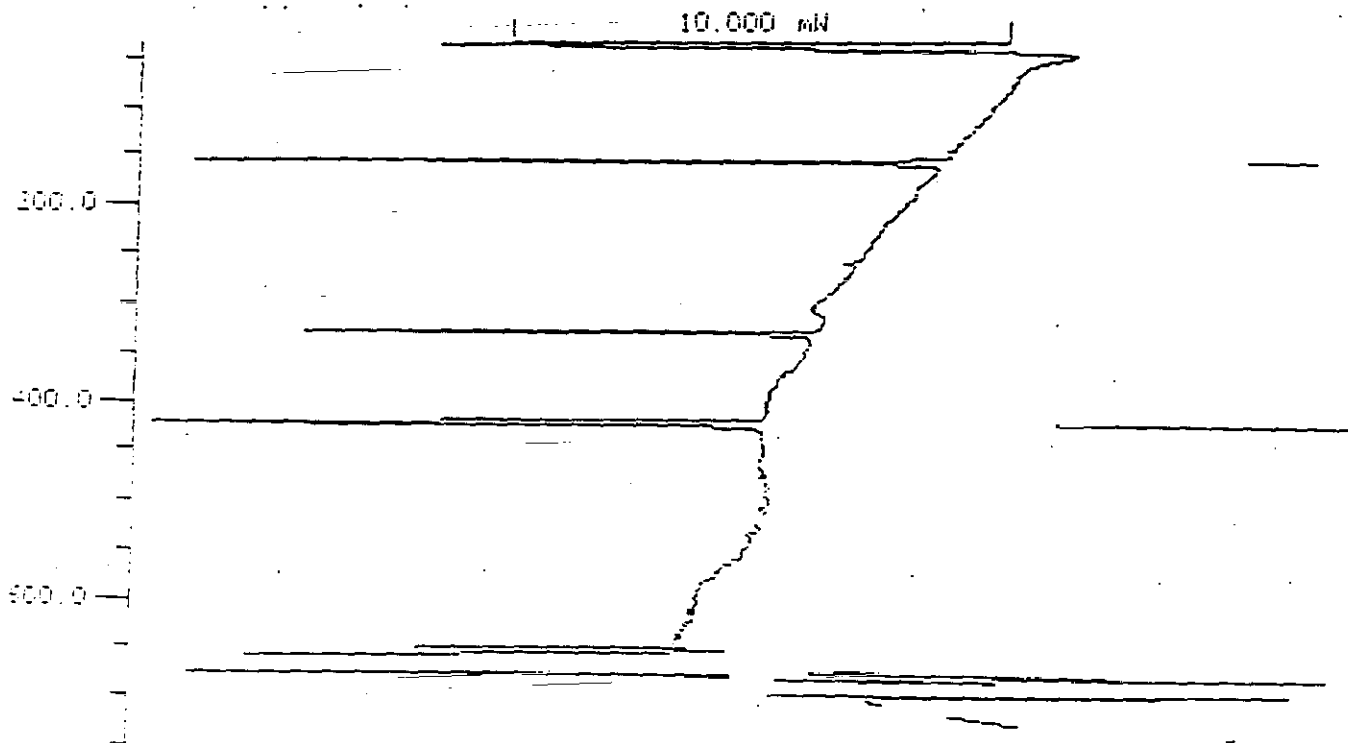
WHC-SD-WM-DP-058

Rev. 0-KB

Jan 31/94

TEMPERATURE °C

HEAT FLOW
EXOTHERMAL-->



A	PT100	.21401
B	PT100	.75726
C	PT100	-.12421
E	INDIUM	310.23

***** METTLER TA4000 SYSTEM *****

Jan R. Laffin
1/31/94

WHC-EP-0806

WHC-SD-WM-DP-058
REV. 0-B

ADDITIONAL DATA SUBMITTED ON 05/18/94

WESTINGHOUSE HANFORD COMPANY

WHC-SD-WM-DP-058

222-S LABORATORY

ANALYTICAL BATCH

REV. 0-~~XB~~Lab Segment Serial No. *2/24/94*
J1668-72Customer ID:
1240-D-A,B,C,D,EAnalysis:
DSCSample Prep:
DIRECT

Instrument: WC16134

Procedure/ Rev: LA 514-113/B-0

Technologist: R. WENDLAND

Date: 05/03/94 *5/2/94*

Starting Time: 0600

Temperature N/A

Ending Time: 2300

Chemist: J. FRYE

Comments:

	Description	Lab ID
1	LMCS	J1667-5511
2	SAMPLE	J1668-5711
3	DUP	J1668-5811
4	SAMPLE	J1669-5711
5	DUP	J1669-5811
6	SAMPLE-- --	J1670-5711
7	DUP	J1670-5811
8	SAMPLE	J1671-5711
9	DUP	J1671-5811
10	SAMPLE	J1672-5711

	Description	Lab ID
11	DUP	J1672-5811
12		
13		
14		
15		
16		
17		
18		
19		
20		

Standard Type	Primary Book No. and Aliquot Vol.	Second Book No. and Aliquot Vol.	Third Book No. and Aliquot Vol.	Final Vol. of Standard
LMCS	12N14-A			

WHC-SD-WM-DP-058

REV. 0-~~AB~~

MUN 82494

34-555-10119-591

DON'T SAY IT ... Write It!

TO Data Handling

FROM

DATE 5/4/94
J. M. LyeBatch 3364

The DSC exotherms in these T-111 samples were very broad. The right end of the exother was easily seen, but the left end merged into the water endotherm. The plots provided are optimized plots. The runs were all done to 500°C.

J. M. Lye
5/4/94



"TO MAKE LIFE LAST, PUT SAFETY FIRST"



WHC-EP-0806

WHC-SD-WM-DP-058

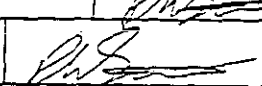
REV. 0-13

for 5/24/94

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

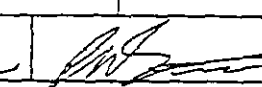
SUPER RUSH

Batch 3364

Serial No J 1667.-5511	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 11:54	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units % RECOVERY	Charge Code N54D2	Reruns 0
Sample Size 6.680 mg			Customer ID STD	
Remarks, Calculations, Results Indium Std. 27.19 g/g 95.3 % Recovery 27.39 g/g 96.0 % Recovery REBEY WENDLAND STD # 12N14-A				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date 5-2-94	Time Completed 23:00	Lab Unit Mgr J.M. Fayer		

54-6800-061 (R-10-83)

SUPER RUSH

Serial No J 1668.-5711	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 11:57	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Reruns 0
Sample Size 16.957 mg			Customer ID 1240-D-A	
Remarks, Calculations, Results 251.2 g/g exotherm REBEY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Fayer		

54-6800-061 (R-10-83)

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WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-18

JWW 5/24/94

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

SUPER RUSH

Serial No	J 1668.-5811	Sample Point	T111-C33-S2	Date	5- 2-94	Time Issued	11:57	Priority	14
Determination	DSC	Method Standard	LA-514-113	Result Units	EXOTHERMS	Charge Code	N54D2	Returns	0
Sample Size	13.267 mg						Customer ID	1240-D-A	
Remarks, Calculations, Results DUPLICATE SAMPLE 269.0 g/g exotherm ROBBY WENDLAND									
Analyst - 1	Analyst - 2		Analyst - 3		Analyst - 4		Analyst - 5		
MIS	MIS		MIS		MIS		MIS		
Date	5-2-93	Time Completed	2500	Lab Unit Mgr	J.M. Fugate		P.W. B...		
5-2-94 RW									

54-6600-061 (R-10-83)

SUPER RUSH

Serial No	J 1669.-5711	Sample Point	T111-C33-S2	Date	5- 2-94	Time Issued	12: 0	Priority	14
Determination	DSC	Method Standard	LA-514-113	Result Units	EXOTHERMS	Charge Code	N54D2	Returns	0
Sample Size	12.576 mg						Customer ID	1240-D-B	
Remarks, Calculations, Results 309.2 g/g exotherm ROBBY WENDLAND									
Analyst - 1	Analyst - 2		Analyst - 3		Analyst - 4		Analyst - 5		
MIS	MIS		MIS		MIS		MIS		
Date	5-2-94	Time Completed	2300	Lab Unit Mgr	J.M. Fugate		P.W. B...		

54-6600-061 (R-10-83)

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WHC-SD-WM-DP-058

REV. 0-XB *Handwritten initials*

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

SUPER RIISH

Serial No J 1669.-5811	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 0	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Retuns 0
Sample Size <i>18.182 mg</i>			Customer ID 1240-D-B	
Remarks, Calculations, Results DUPLICATE SAMPLE <i>287.5 g/g exotherm</i> <i>ROBBY WENDLAND</i>				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mrs	Mrs	Mrs	Mrs	Mrs
Date 5-2-94	Time Completed 2300	Lab Unit Mgr <i>J. M. Luy</i>	<i>[Signature]</i>	

54-6800-061 (R-10-E3)

SUPER RIISH

Serial No J 1670.-5711	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 1	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Retuns 0
Sample Size <i>5.897 mg</i>			Customer ID 1240-D-C	
Remarks, Calculations, Results <i>180.2 g/g exotherm</i> <i>ROBBY WENDLAND</i>				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mrs	Mrs	Mrs	Mrs	Mrs
Date 5-2-94	Time Completed 2300	Lab Unit Mgr <i>J. M. Luy</i>	<i>[Signature]</i>	

54-6800-061 (R-10-E3)

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WHC-SD-WM-DP-058

Rev. 0-1B *HW 5/24/94*

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

SUPER RUSH

Serial No.	Sample Point	Date	Time Issued	Priority
J 1670.-5811	T111-C33-S2	5- 2-94	12: 1	14
Determination	Method Standard	Result Units	Charge Code	Returns
DSC	LA-514-113	EXOTHERMS	N54D2	0
Sample Size			Customer ID	
6.179 mg			1240-D-C	
Remarks, Calculations, Results				
DUPLICATE SAMPLE				
187.1 g/g exotherm				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mrs	Mrs	Mrs	Mrs	Mrs
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Lynn	<i>[Signature]</i>	

54-6600-061 (R-10-83)

GUIDED DISCU

Serial No.	Sample Point	Date	Time Issued	Priority
J 1671.-5711	T111-C33-S2	5- 2-94	12: 2	14
Determination	Method Standard	Result Units	Charge Code	Returns
DSC	LA-514-113	EXOTHERMS	N54D2	0
Sample Size			Customer ID	
6.696 mg			1240-D-D	
Remarks, Calculations, Results				
162.7 g/g exotherm				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mrs	Mrs	Mrs	Mrs	Mrs
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Lynn	<i>[Signature]</i>	

54-6600-061 (R-10-83)

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WHC-EP-0806

WHC-SD-WM-DP-058

Rev. O-KB *HW 5/24/94*

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

SUPER RICH				
Serial No. J 1671.-5811	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 2	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Reruns 0
Sample Size 7.023 mg			Customer ID 1240-D-D	
Remarks, Calculations, Results DUPLICATE SAMPLE 175.3 g/g exotherm Robby Wendland				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MS	MS	MS	MS	MS
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Lye <i>[Signature]</i>		

54-6600-061 (R-10-23)

SUPER RICH				
Serial No. J 1672.-5711	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 3	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Reruns 0
Sample Size 7.740 mg			Customer ID 1240-D-E	
Remarks, Calculations, Results 336.2 g/g exotherm Robby Wendland				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MS	MS	MS	MS	MS
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Lye <i>[Signature]</i>		

54-6600-061 (R-10-23)

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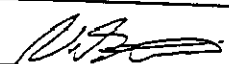
WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-1/5

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS - UNDIGESTED SAMPLE

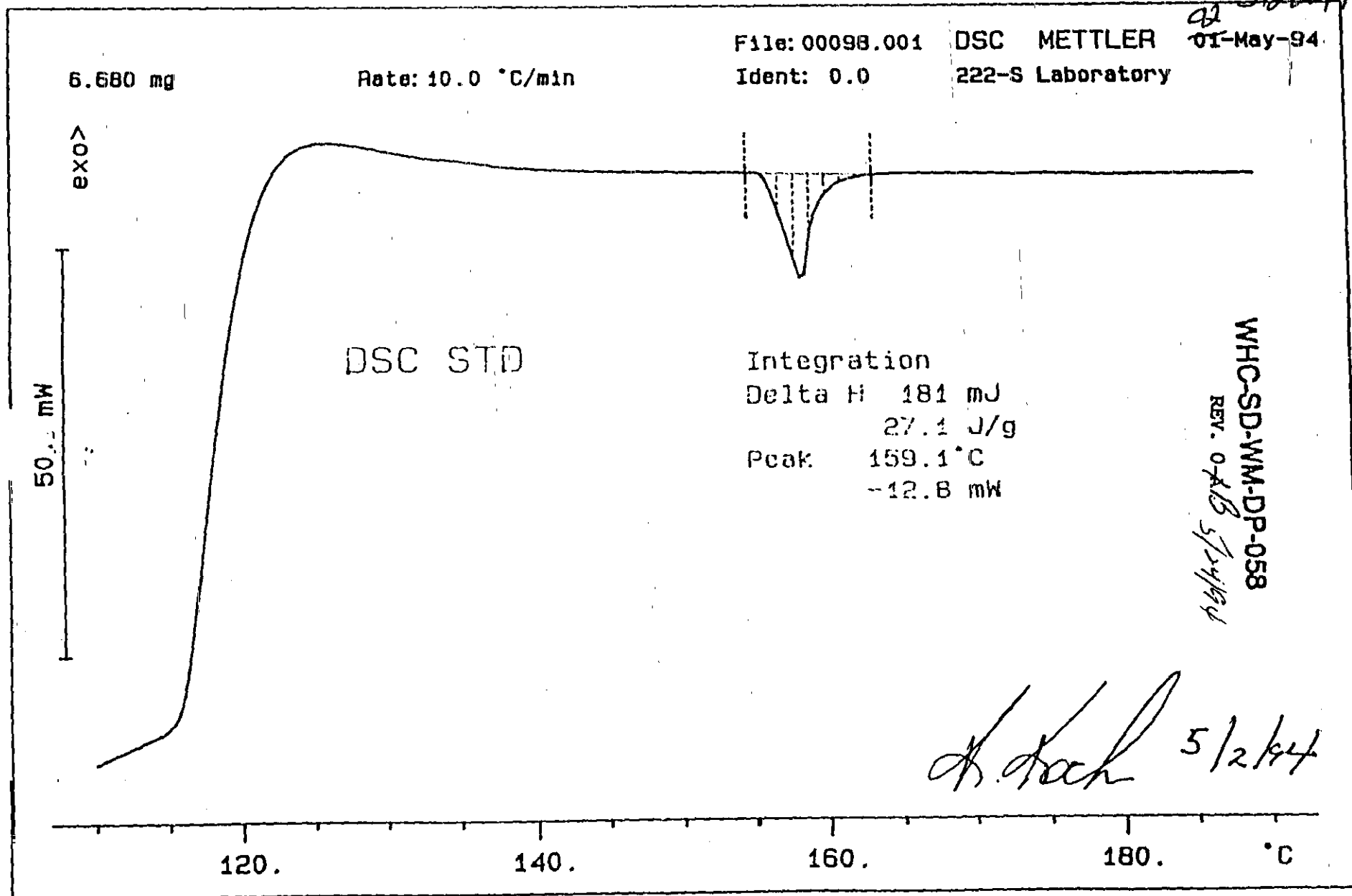
SUPER RUSH

Serial No J 1672.-5811	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 3	Priority 14
Determination DSC	Method Standard LA-514-113	Result Units EXOTHERMS	Charge Code N54D2	Returns 0
Sample Size 6.154 mg			Customer ID 1240-D-E	
Remarks, Calculations, Results DUPLICATE SAMPLE 335.9 J/g exotherm ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date 5-2-94	Time Completed 2300	Lab Unit 1/2/3/4 J.M. Jager		

54-8890-061 (R-10-83)

BEST AVAILABLE COPY

Signature below represents chemical technologist/chemist that completed/verified the calibration/analysis on pages 105 to 116.



DSC STD

6.680 mg

Rate: 10.0 °C/min

File: 00109.001

DSC METTLER

02-May-94

Ident: 0.0

222-S Laboratory

exo

DSC STD

Integration

Delta H 183 mJ

27.3 J/g

Peak 159.1 °C

-13.0 mW

WHC-SD-WM-DP-058

REV. 0-25

5/24/94 (ML)

WHC-EP-0806

120.

140.

160.

180.

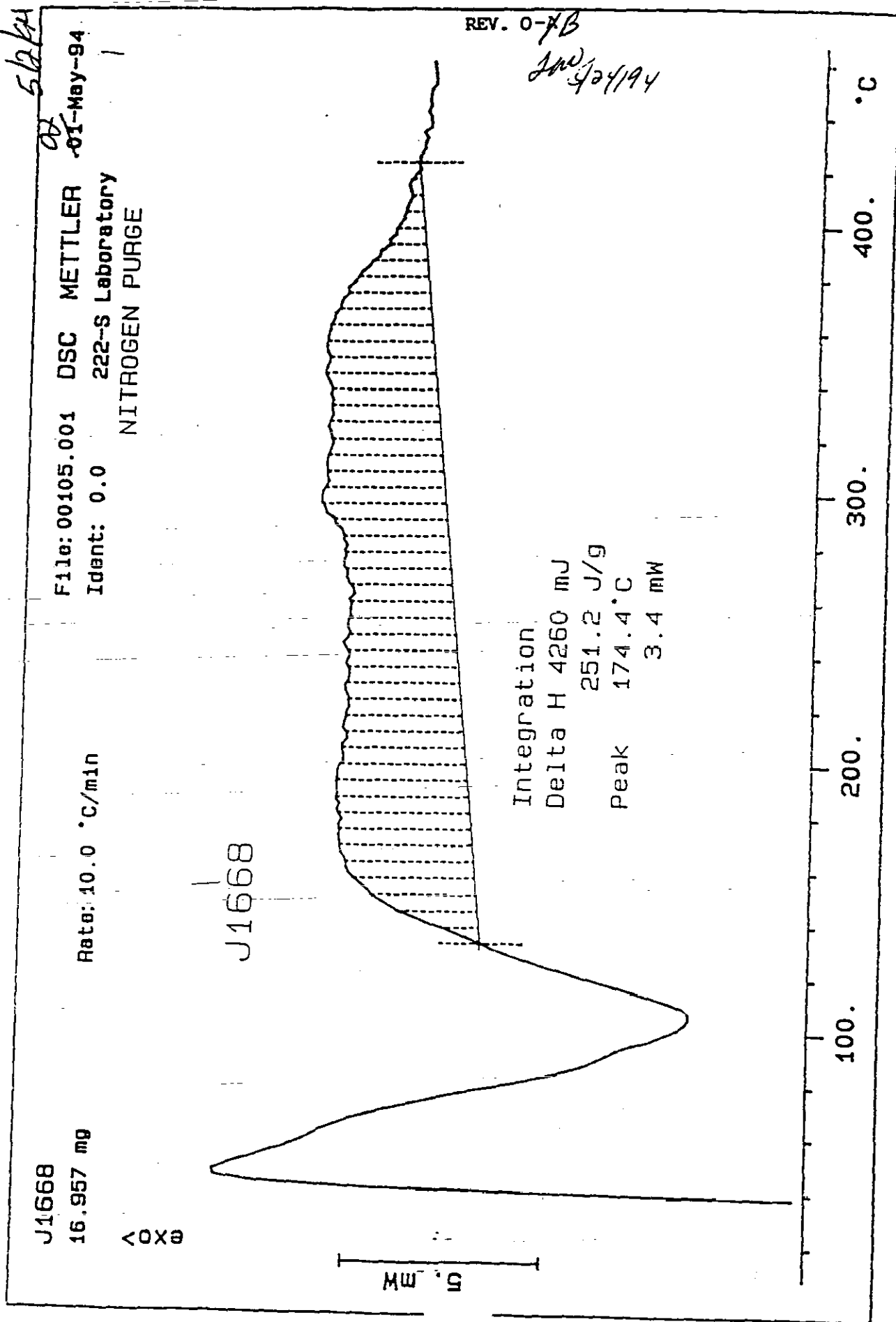
°C

10.0 mW

A-142

REV. 0-XB

JAN 24 1994



J1668 DUP

13.267 mg

Rate: 10.0 °C/min

File: 00107.001

DSC METTLER

02 *5/2/94*
01-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo >

J1668 DUP

MW - 0.01

Integration

Delta H 3568 mJ

269.0 J/g

Peak 188.4 °C

3.1 mW

100.

200.

300.

400.

°C

WHC-SD-WM-DP-058

WHC-EP-0806

REV. 0-1/8 *5/2/94 AW*

J1669

12.596 mg

Rate: 10.0 °C/min

File: 00119.001 DSC METTLER 02-May-94

Ident: 0.0 222-S Laboratory

NITROGEN PURGE

exo

J1669

10.0 mW

A-145

Integration
Delta H 3894 mJ
309.2 J/g
Peak 296.7 °C
3.0 mW

REV. 0-15

Handwritten signature

WHI-SD-YM-DF-058

MHC-EP-0806

100.

200.

300.

400.

°C

5/5/94
89

J1689 DUP

18.182 mg

Rate: 10.0 °C/min

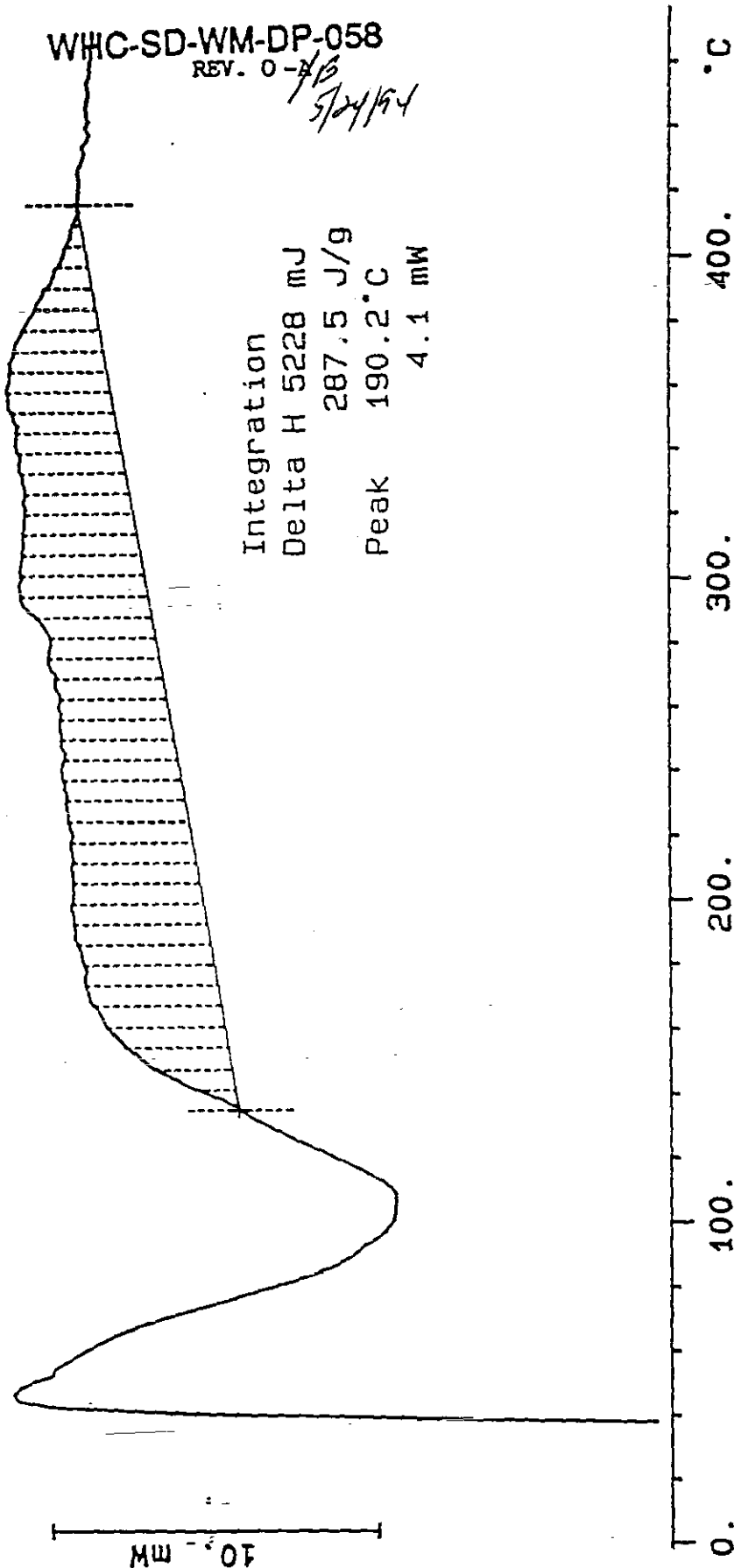
File: 00121.001 DSC METTLER 02-May-94

Ident: 0.0 222-S Laboratory

NITROGEN PURGE

J1669 DUP

<exo>



J1670
5.897 mg

Rate: 10.0 °C/min

File: 00112.001 DSC METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo

J1670

5 mW

Integration
Delta H 1063 mJ
180.2 J/g
Peak 302.7 °C
0.9 mW

WHC-SD-WM-DP-058
REV 1 0-1/93
3/24/94

WHC-EP-0806

A-147

100.

200.

300.

400.

°C

J1670 DUP

6.179 mg

Rate: 10.0 °C/min

File: 00113.001 DSC METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo

J1670 DUP

WHC-SD-WM-DP-058

REV. 0-4/95

5/24/95

5 mW

Integration
Delta H 1156 mJ
187.1 J/g
Peak 296.8 °C
1.1 mW

100.

200.

300.

400.

°C

WHC-EP-0806

J1671
6.696 mg

Rate: 10.0 °C/min

File: 00103.001 DSC METTLER
Ident: 0.0 222-S Laboratory

02 5/2/94 KL
01-May-94

NITROGEN PURGE

< exo

J1671

WHC-SD-WM-DP-058
REV. 0-1/15

WHC-EP-0806

Integration
Delta H 1090 mJ
162.7 J/g
Peak 302.8 °C
1.0 mW

MW 5

A-149

100. 200. 300. 400. °C

J1671 DUP

7.023 mg

Rate: 10.0 °C/min

File: 00111.001

DSC METTLER

02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo
end

J1671 DUP

WHC-SD-WM-DP-058

REV. 0-1-88

SN 1404154

Integration
Delta H 1231 mJ
175.3 J/g
Peak 302.9°C
1.0 mW

5 mW

100.

200.

300.

400.

°C

J1672

7.740 mg

Rate: 10.0 °C/min

File: 00115.001

DSC METTLER

02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo

J1672

WHC-SD-WM-DP-058

REV. 0-4/95

5/14/95

5 mW

Integration
Delta H 2603 mJ
336.3 J/g
Peak 298.8 °C
2.1 mW

100.

200.

300.

°C

J1672 DUP

6.154 mg

Rate: 10.0 °C/min

File: 00117.001

DSC METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

exo
^

J1672 DUP

WHC-SD-WM-DP-058

REV. 04/8

5/24/91

10. mW

Integration

Delta H 2067 mJ

335.9 J/g

Peak 184.4 °C

1.6 mW

100.

200.

300.

400.

°C

WHC-EP-0806

A-152

WESTINGHOUSE HANFORD COMPANY

WHC-SD-WM-DP-058

222-S LABORATORY

REV. 0-*XB* *HW*

ANALYTICAL BATCH

Lab Segment Serial No.

J1668-72

Customer ID:

1240-D-A,B,C,D,E

Analysis:

TGA

Sample Prep:

DIRECT

Instrument: WC16130

Procedure/ Rev: LA 560-112/A-1

Technologist: R. WENDLAND

Date: 05/03/94 *02/11/94* *5/2/94*

Starting Time: 600

Temperature N/A

Ending Time: 2300

Chemist: J. FRYE

Comments:

	Description	Lab ID
1	LMCS	J1667-5512
2	SAMPLE	J1668-5712
3	DUP	J1668-5812
4	SAMPLE	J1669-5712
5	DUP	J1669-5812
6	SAMPLE	J1670-5712
7	DUP	J1670-5812
8	SAMPLE	J1671-5712
9	DUP	J1671-5812
10	SAMPLE	J1672-5712

	Description	Lab ID
11	DUP	J1672-5812
12		
13		
14		
15		
16		
17		
18		
19		
20		

Standard Type	Primary Book No. and Aliquot Vol.	Second Book No. and Aliquot Vol.	Third Book No. and Aliquot Vol.	Final Vol. of Standard
LMCS	24N8-A			

WHC-SD-WM-DP-058

REV. 0-13 5/24/94

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

SUPER RUSH

Batch 3365

Serial No	Sample Point	Date	Time Issued	Priority
J 1667.-5512	T111-C33-S2	5- 2-94	11:54	14
Determination	Method Standard	Result Units	Charge Code	Returns
TGA	LA-560-112	% RECOVERY	N54D2	0
Sample Size	Customer ID			
16.955 mg	STD			
Remarks, Calculations, Results				
58.7% H ₂ O 99.1% Recovery				
Robby Wendland STD# 2418-A				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
M/S	M/S	M/S	M/S	M/S
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Faye	P.B.	

54-6800-061 (R-10-E31)

GUIDED RUSH

Serial No	Sample Point	Date	Time Issued	Priority
J 1668.-5712	T111-C33-S2	5- 2-94	11:57	14
Determination	Method Standard	Result Units	Charge Code	Returns
TGA	LA-560-112	% H ₂ O	N54D2	0
Sample Size	Customer ID			
11.928 mg	1240-D-A			
Remarks, Calculations, Results				
11.68% H ₂ O				
Robby Wendland				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
M/S	M/S	M/S	M/S	M/S
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Faye	P.B.	

54-6800-061 (R-10-E31)

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WHC-SD-WM-DP-058

REV. 0-AB 5/24/94

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

CLIPER RUSH

Serial No	Sample Point	Date	Time Issued	Priority
J 1668.-5812	T111-C33-S2	5- 2-94	11:57	14
Determination	Method Standard	Result Units	Charge Code	Returns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
15.604 mg			1240-D-A	
Remarks, Calculations, Results				
DUPLICATE SAMPLE				
10.06 % H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J. M. Faye	[Signature]	

54-6800-061 (R-10-83)

CLIPER RUSH

Serial No	Sample Point	Date	Time Issued	Priority
J 1669.-5712	T111-C33-S2	5- 2-94	12: 0	14
Determination	Method Standard	Result Units	Charge Code	Returns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
17.548 mg			1240-D-B	
Remarks, Calculations, Results				
10.66 % H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J. M. Faye	[Signature]	

54-6800-061 (R-10-83)

BEST AVAILABLE COPY

WHC-EP-0806

WHC-SD-WM-DP-058

REV. 0-7B 5/24/94 JAW

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

SUPER RUSH

Serial No.	Sample Point	Date	Time Issued	Priority
J 1669.-5812	T111-C33-S2	5- 2-94	12: 0	14
Determination	Method Standard	Result Units	Charge Code	Retuns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
19.760 mg			1240-D-B	
Remarks, Calculations, Results				
DUPLICATE SAMPLE				
9.90% H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Faye	RWB	

54-6600-061 (R-10-83)

SUPER RUSH

Serial No.	Sample Point	Date	Time Issued	Priority
J 1670.-5712	T111-C33-S2	5- 2-94	12: 1	14
Determination	Method Standard	Result Units	Charge Code	Retuns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
6.338 mg			1240-D-C	
Remarks, Calculations, Results				
11.35% H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MIS	MIS	MIS	MIS	MIS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J.M. Faye	RWB	

54-6600-061 (R-10-83)

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WHC-SD-WM-DP-058

REV. 0-2/15 MW 5/24/94

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

SUPER RUSH

Serial No J 1670.-5812	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 1	Priority 14
Determination TGA	Method Standard LA-560-112	Result Units % H2O	Charge Code N54D2	Returns 0
Sample Size 8.043 mg			Customer ID 1240-D-C	
Remarks, Calculations, Results DUPLICATE SAMPLE 11.99 % H ₂ O Robby Wendland				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MISS	MISS	MISS	MISS	MISS
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Fayer	Signature [Signature]	

54-6800-061 (R-10-E3)

SUPER RUSH

Serial No J 1671.-5712	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 2	Priority 14
Determination TGA	Method Standard LA-560-112	Result Units % H2O	Charge Code N54D2	Returns 0
Sample Size 7.786 mg			Customer ID 1240-D-D	
Remarks, Calculations, Results 12.07 % H ₂ O Robby Wendland				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MISS	MISS	MISS	MISS	MISS
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Fayer	Signature [Signature]	

54-6800-061 (R-10-E3)

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WHC-SD-WM-DP-058

Rev. 0-1/8

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

GUIDED DISCU

Serial No.	Sample Point	Date	Time Issued	Priority
J 1671.-5812	T111-C33-S2	5- 2-94	12: 2	14
Determination	Method Standard	Result Units	Charge Code	Retuns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
5.750 mg			1240-D-D	
Remarks, Calculations, Results				
DUPLICATE SAMPLE				
12.02% H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MISS	MISS	MISS	MISS	MISS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J. M. Luge		

54-6830-061 (R-10-83)

GUIDED DISCU

Serial No.	Sample Point	Date	Time Issued	Priority
J 1672.-5712	T111-C33-S2	5- 2-94	12: 3	14
Determination	Method Standard	Result Units	Charge Code	Retuns
TGA	LA-560-112	% H2O	N54D2	0
Sample Size			Customer ID	
7.101 mg			1240-D-E	
Remarks, Calculations, Results				
11.21% H ₂ O				
ROBBY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
MISS	MISS	MISS	MISS	MISS
Date	Time Completed	Lab Unit Mgr		
5-2-94	2300	J. M. Luge		

54-6830-061 (R-10-83)

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WHC-SD-WM-DP-058

REV. 0-1B

5/24/94 New

THERMAL GRAVIMETRIC ANALYSIS - UNDIGESTED SAMPLE

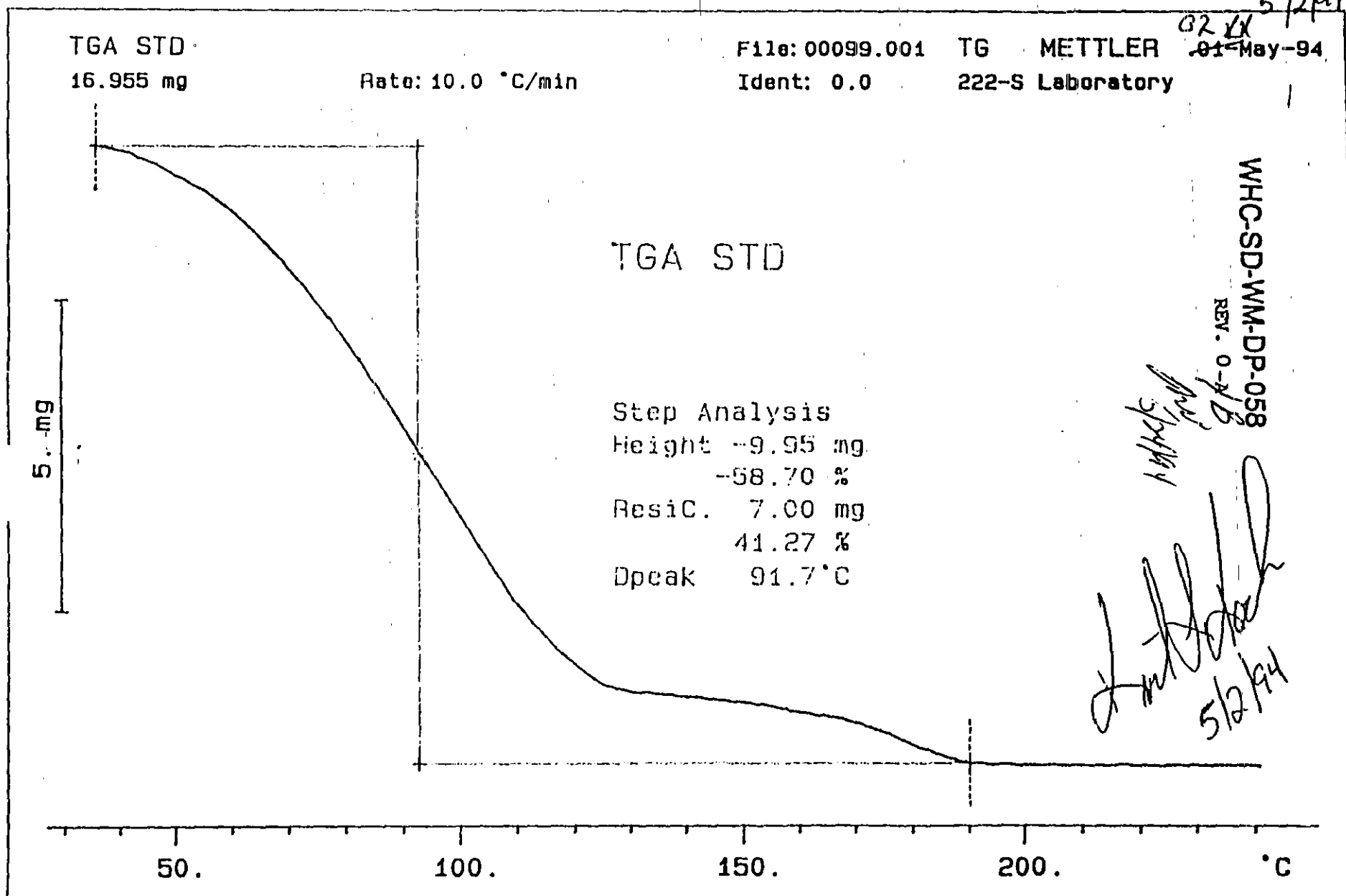
SUPER RIISH

Serial No. J 1672.-5812	Sample Point T111-C33-S2	Date 5- 2-94	Time Issued 12: 3	Priority 14
Determination TGA	Method Standard LA-560-112	Result Units % H2O	Charge Code N54D2	Retuns 0
Sample Size 9.313 mg			Customer ID 1240-D-E	
Remarks, Calculations, Results DUPLICATE SAMPLE 11.10% H ₂ O ROSEY WENDLAND				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Date 5-2-94	Time Completed 2300	Lab Unit Mgr J.M. Luge	[Signature]	

54 6800-061 R-10-821

BEST AVAILABLE COPY

Signature below represents chemical Technologist/chemist that completed/verified the calibration/analysis on page 124 to 135.



TGA STD

12.818 mg

Rate: 10.0 °C/min

File: 00110.001

TG

METTLER

02-May-94

Ident: 0.0

222-S Laboratory

TGA STD

Step Analysis

Height -7.60 mg

-59.25 %

ResidC. 5.22 mg

40.70 %

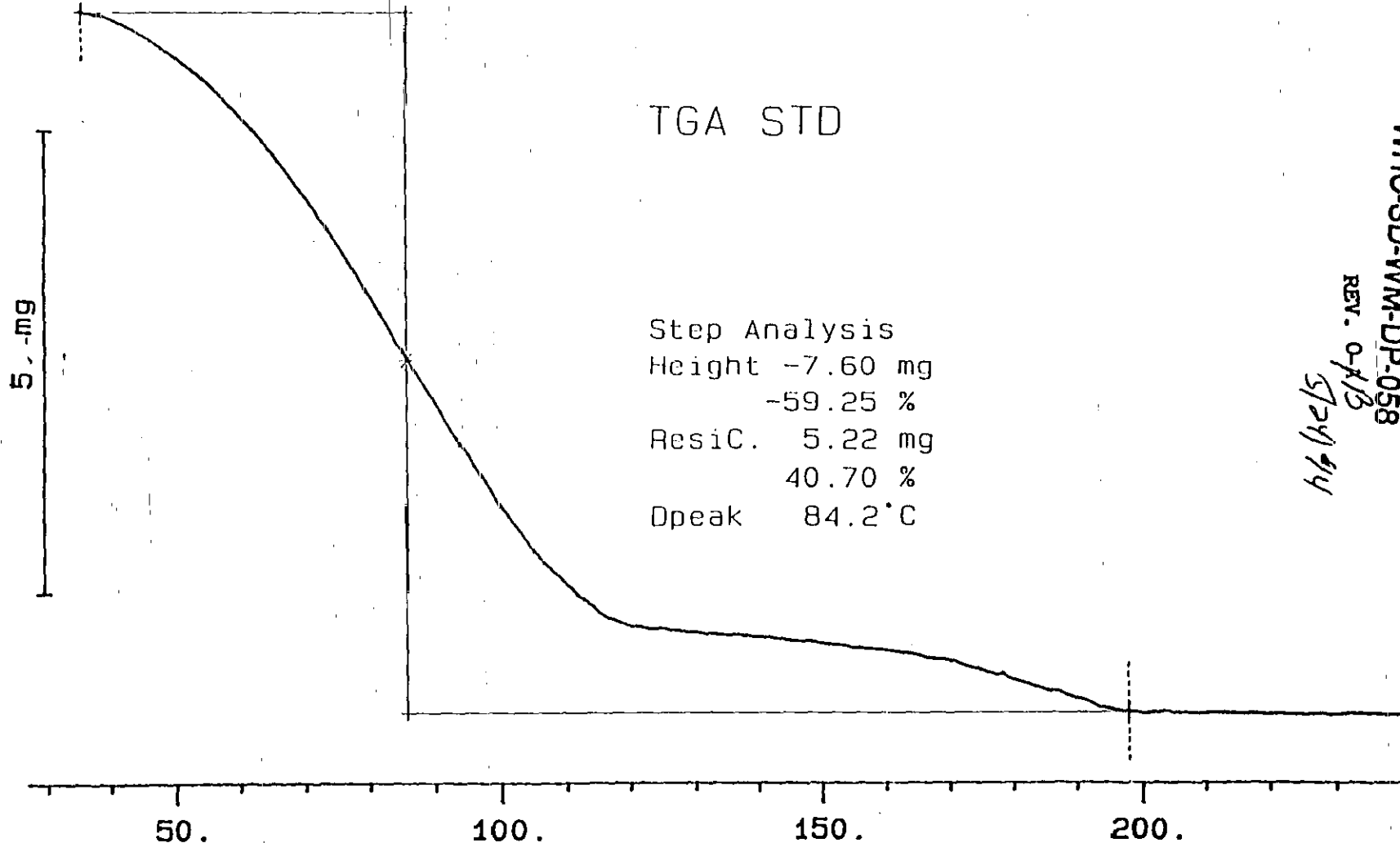
Dpeak 84.2 °C

WHC-SD-WM-DP-058

REV. 0-1/8

5/24/94

WHC-EP-0806



50.

100.

150.

200.

°C

A-161

J1668

11.928 mg

Rate: 10.0 °C/min

File: 00104.001

TG

METTLER

01-May-94

Ident: 0.0

222-S Laboratory

1/2 page 5/2/94

Step Analysis

Height -1.39 mg

-11.68 %

ResidC. 10.53 mg

88.30 %

Dpeak 67.0 °C

J1668

WHC-SD-WM-DP-058

REV. 0-1/8

5/24/94

WHC-EP-0806

A-162

1.39 mg

100. 200. 300. 400. °C

J1668 DUP

15.604 mg

Rate: 10.0 °C/min

File: 00106.001

TG

METTLER

02-5/2/94
01-May-94

Ident: 0.0

222-S Laboratory

N2 purge 5/5/94

Step Analysis

Height -1.57 mg

-10.06 %

Resid. 14.03 mg

89.93 %

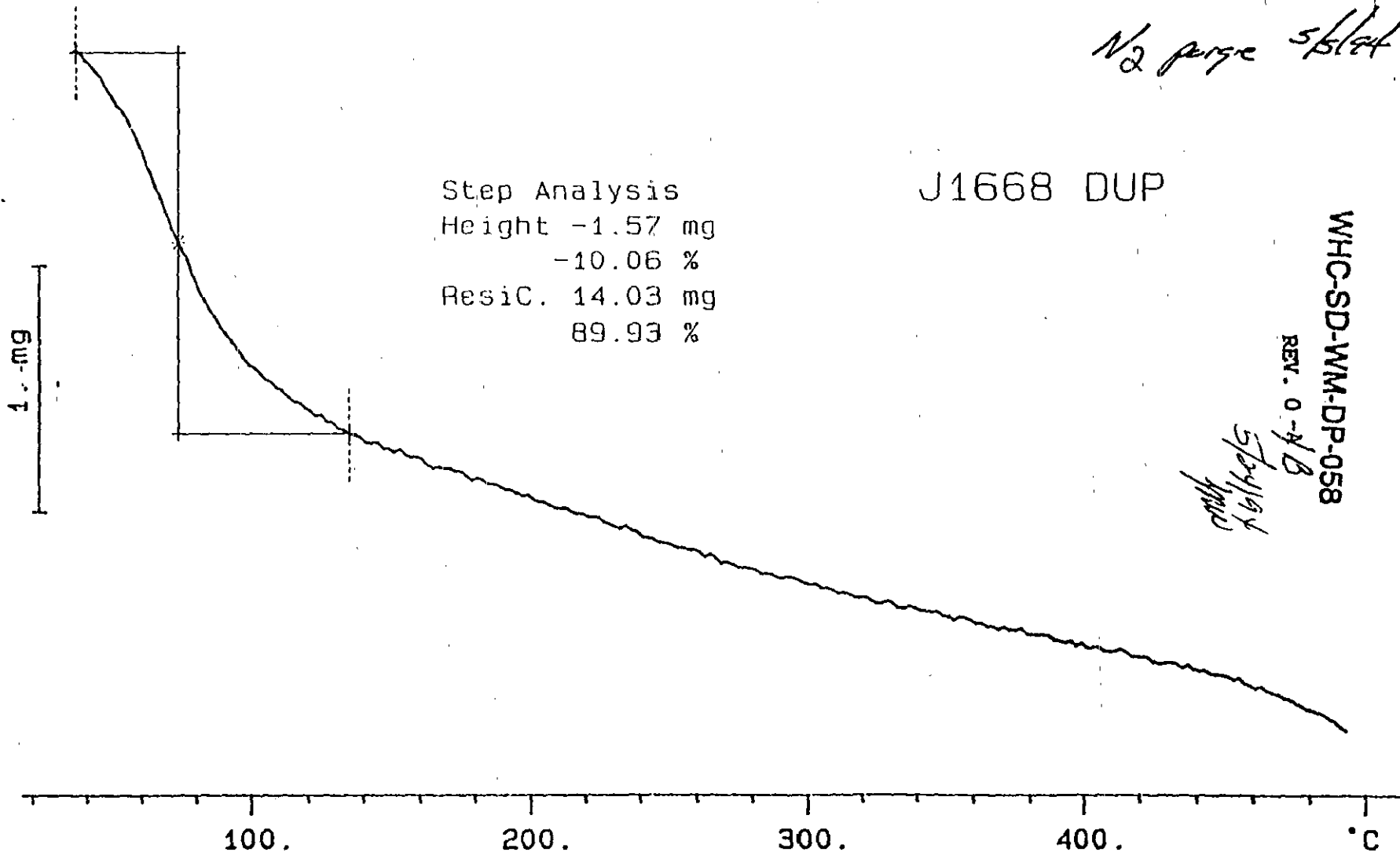
J1668 DUP

WHC-SD-WM-DP-058

REV. 0-1/8

*5/24/94
JWC*

WHC-EP-0806



A-163

J1669

17.548 mg

Rate: 10.0 °C/min

File: 00118.001

TG

METTLER

02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURG

J1669

Step Analysis

Height -1.87 mg

-10.66 %

ResidC. 15.67 mg

89.27 %

WHC-SD-WM-DP-058

REV. 0-1

5/21/94
JMD

WHC-EP-0806

100.

200.

300.

400.

°C

A-164

6W-1

J1669 DUP

19.760 mg

Rate: 10.0 °C/min

File: 00120.001 TG METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

Step Analysis

Height -1.96 mg

-9.90 %

ResidC. 17.80 mg

90.06 %

J1669 DUP

WMC-SD-WM-DP-058

REV. 0-7/94

5/24/94 JWC

6w. 2

A-165

WMC-EP-0806

100.

200.

300.

400.

°C

J1670

6.338 mg

Rate: 10.0 °C/min

File: 00114.001

TG

METTLER

02-May-94

Ident: 0.0

222-S Laboratory

1/2 page 5/5/94

Step Analysis

Height -0.72 mg

-11.35 %

ResiC. 5.62 mg

88.66 %

Dpeak 63.0 °C

J1670

WHC-SD-WM-DP-058

REV. 0.1

5/5/94

A-166

0.5 mg

100. 200. 300. 400. °C

WHC-EP-0806

J1670 DUP

8.043 mg

Rate: 10.0 °C/min

File: 00116.001 TG METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

Step Analysis

Height -0.96 mg

-11.99 %

ResidC. 7.07 mg

87.94 %

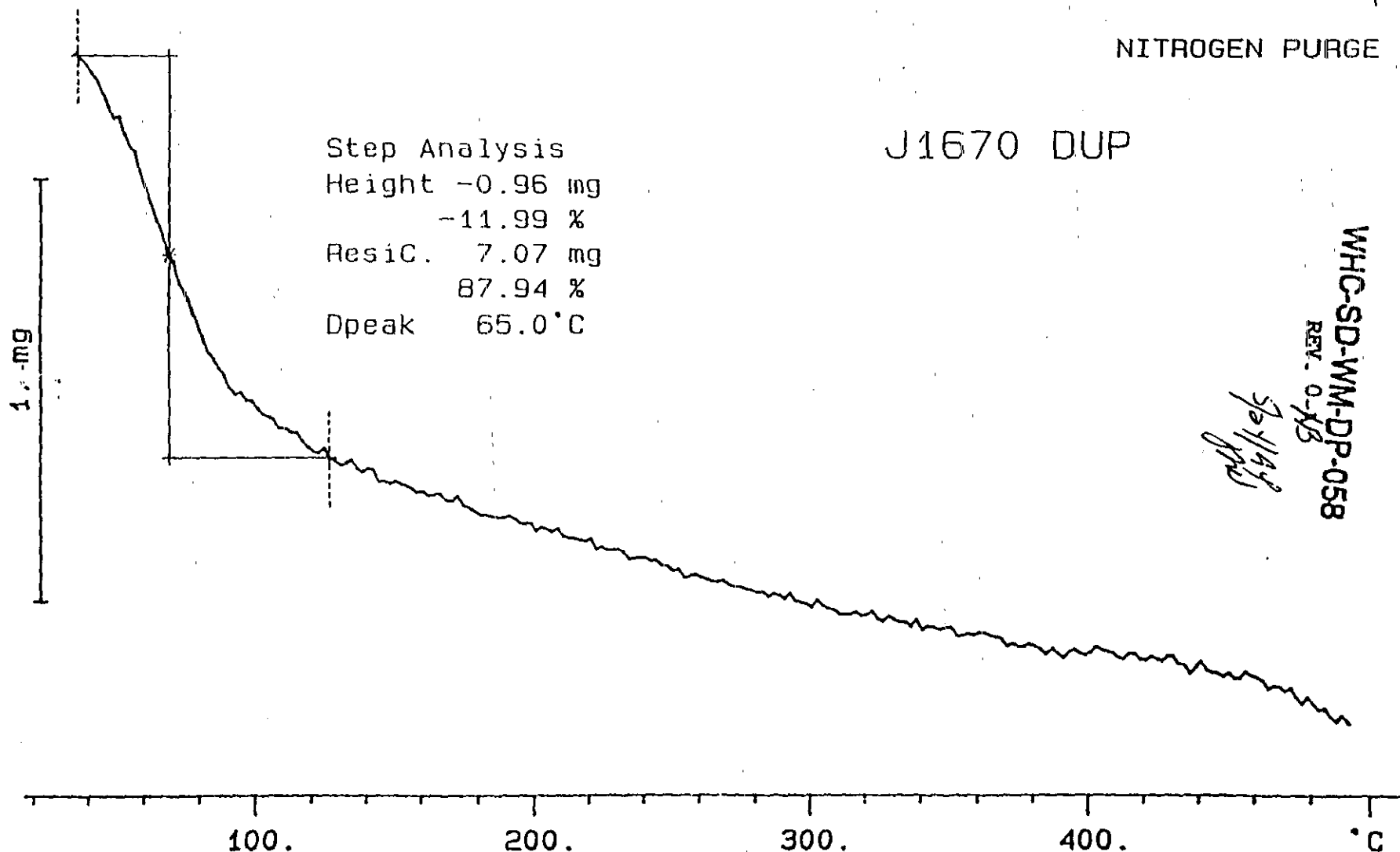
Dpeak 65.0 °C

J1670 DUP

WHC-SD-WM-DP-058

REV. 0-1/85

5/1/94
gpc



A-167

WHC-EP-0806

J1671

7.786 mg

Rate: 10.0 °C/min

File: 00102.001

TG

METTLER

01-May-94

Ident: 0.0

222-S Laboratory

1/2 page 5/5/94

08 5/2/94
✓

Step Analysis

Height -0.94 mg

-12.07 %

ResidC. 6.84 mg

87.87 %

Dpeak 67.0 °C

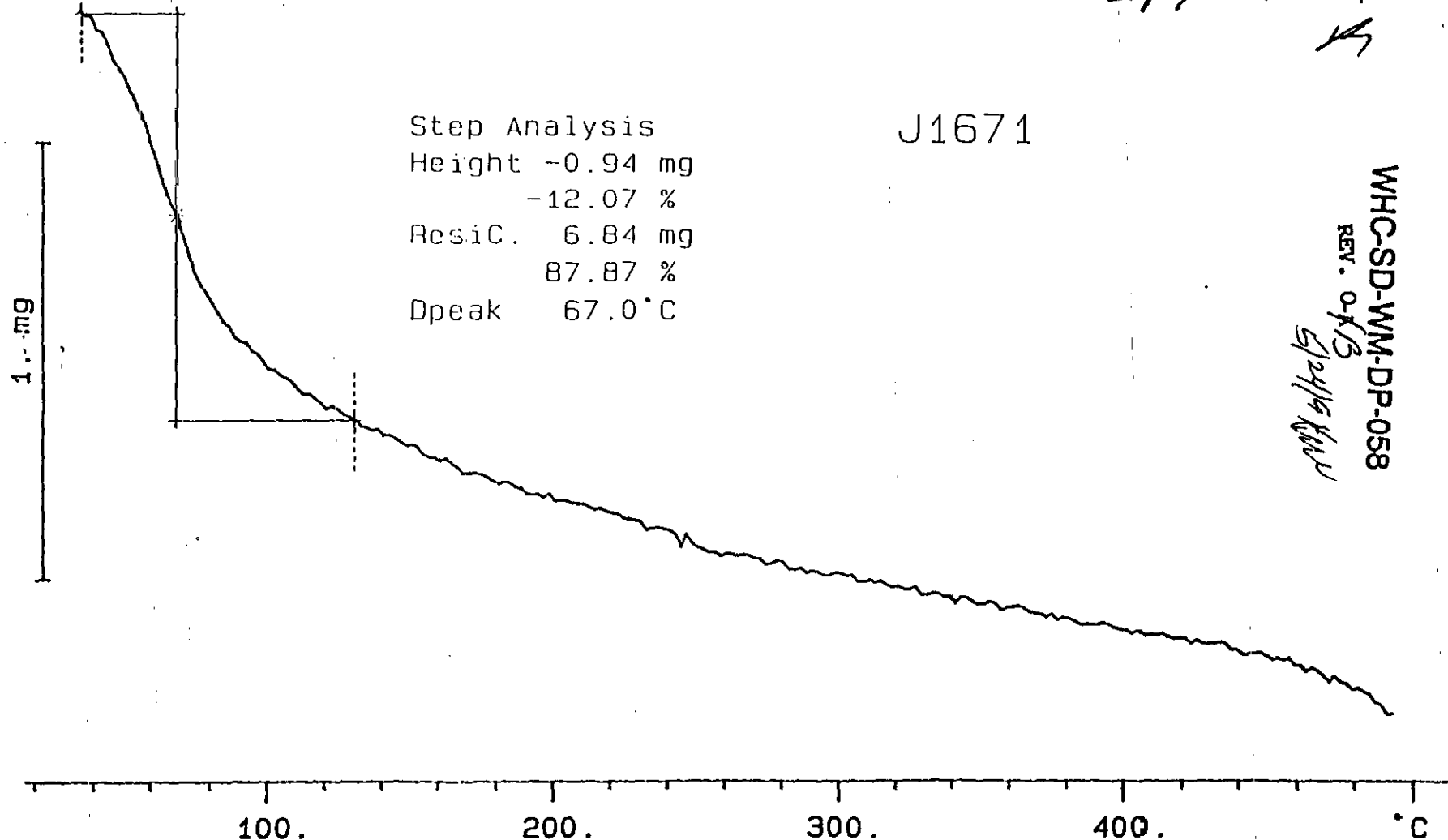
J1671

WHCSD-WM-DP-058

REV. 0.1/13

5/24/94 KAC

WHC-EP-0806



J1671 DUP

5.750 mg

Rate: 10.0 °C/min

File: 00108.001

TG

METTLER

01-May-94

Ident: 0.0

222-S Laboratory

N₂ purge 5/5/94

Step Analysis

Height -0.69 mg

-12.08 %

ResiC. 5.06 mg

87.93 %

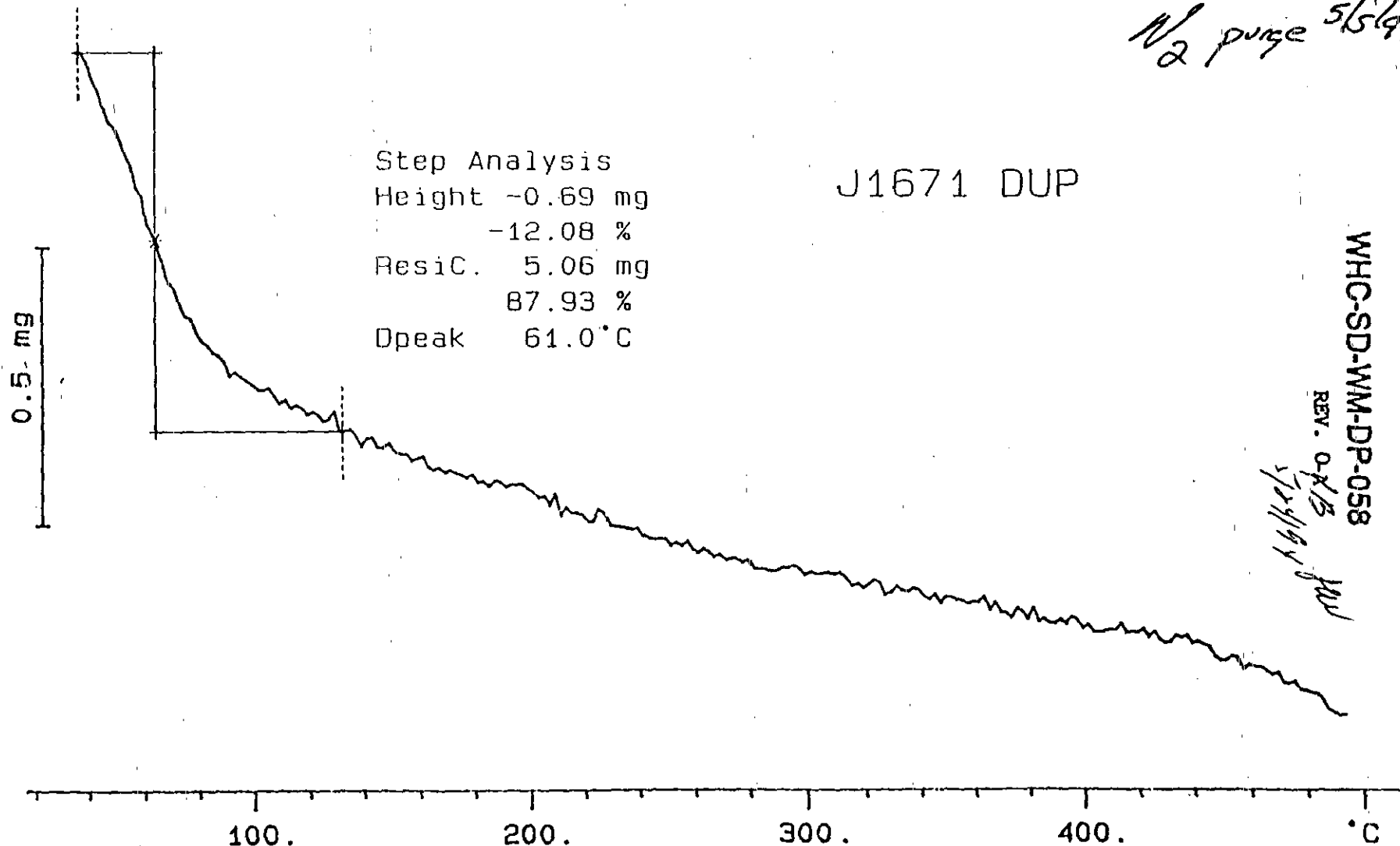
Dpeak 61.0 °C

J1671 DUP

WHC-SD-WM-DP-058

REV. 0-1/5

5/24/94



J1672

7.101 mg

Rate: 10.0 °C/min

File: 00122.001

TG

METTLER

02-May-94

Ident: 0.0

222-S Laboratory

N₂ purge 5/5/94
✓

Step Analysis

Height -0.80 mg

-11.21 %

ResiC. 6.30 mg

88.68 %

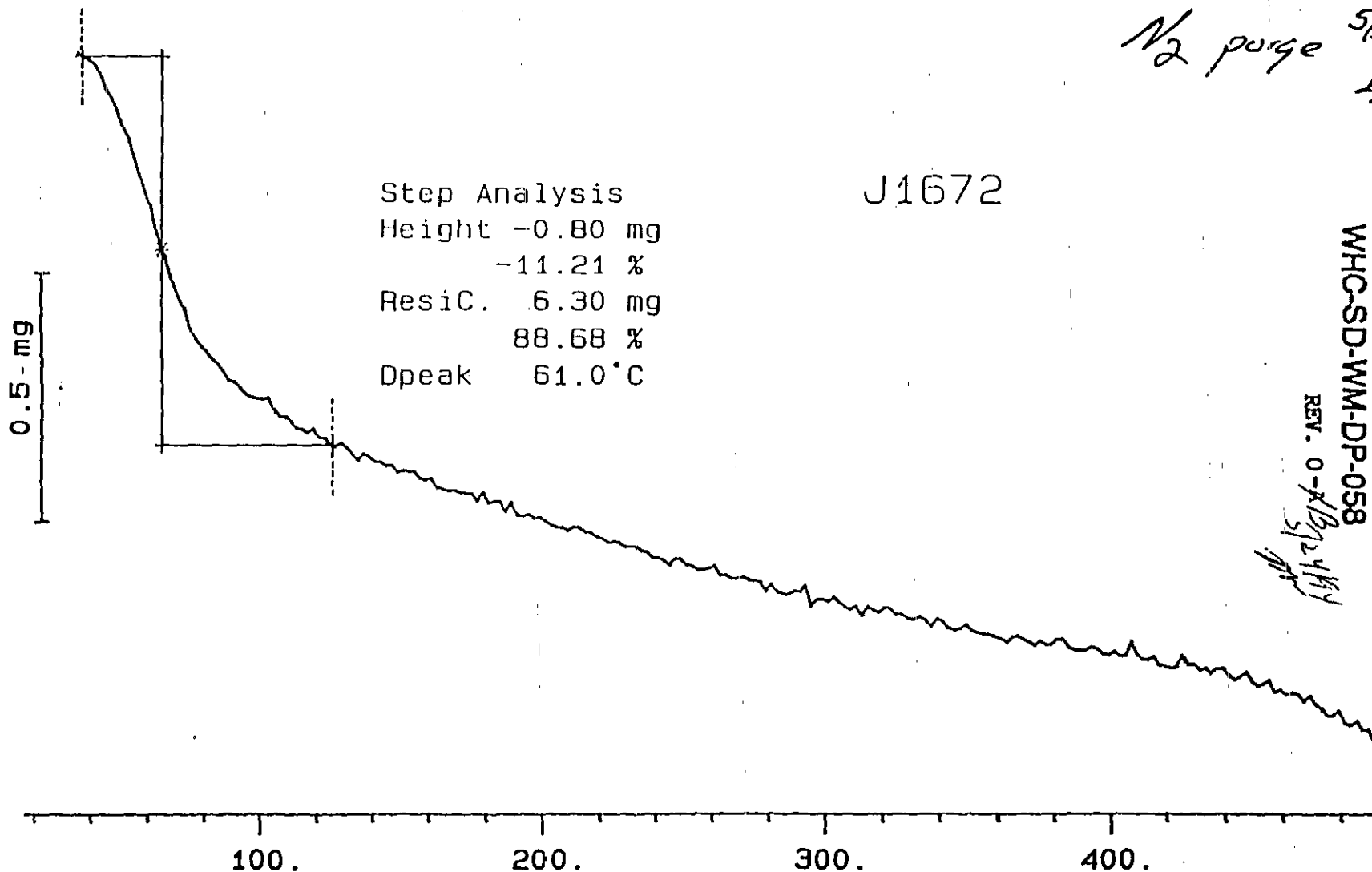
Dpeak 61.0 °C

J1672

WHC-SD-WM-DP-058

REV. 0-1/13/94

5/12/94
✓



J1672 DUP

9.313 mg

Rate: 10.0 °C/min

File: 00123.001 TG METTLER 02-May-94

Ident: 0.0

222-S Laboratory

NITROGEN PURGE

Step Analysis

Height -1.03 mg

-11.10 %

ResiC. 8.28 mg

88.90 %

Dpeak 65.0 °C

J1672 DUP

WHC-SD-WM-DP-058

REV. 0-2/13

Handwritten signature and date: 4/10/94

WHC-EP-0806

100.

200.

300.

400.

°C

6w-1

A-171

WHC-EP-0806

Appendix B: Statistical Interpretation

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SUPPORTING DOCUMENT

1. Total Pages 61

2. Title Statistical Characterization Report for Single-Shell Tank 241-T-111	3. Number WHC-SD-WM-TI-650	4. Rev No. 0
5. Key Words Single-Shell Tank, Waste, Characterization, Sampling, Sample Homogenization, Core Composite Samples <div style="text-align: center;"> APPROVED FOR PUBLIC RELEASE </div> <i>XMB 9/20/94</i>	6. Author Name: R. D. Cromar/S.R. Wilmarth <i>R.D. Cromar Steven R. Wilmarth</i> Signature Name: Louis Jensen <i>L. Jensen 9/20/94</i> Signature Organization/Charge Code 8E100/N4D2G	
7. Abstract This report documents the statistical analyses performed on core data from Single-Shell Tank 241-T-111		
8. PURPOSE AND USE OF DOCUMENT - This document was prepared for use within the U.S. Department of Energy and its contractors. It is to be used only to perform, direct, or integrate work under U.S. Department of Energy contracts. This document is not approved for public release until reviewed. PATEM STATUS - This document copy, since it is transmitted in advance of patent clearance, is made available in confidence solely for use in performance of work under contracts with the U.S. Department of Energy. This document is not to be published nor its contents otherwise disseminated or used for purposes other than specified above before patent approval for such release or use has been secured, upon request, from the Patent Counsel, U.S. Department of Energy Field Office, Richland, WA. DISCLAIMER - This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or any third party's use or the results of such use of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.	10. RELEASE STAMP <div style="border: 1px solid black; padding: 10px; text-align: center;"> OFFICIAL RELEASE 5 BY WHC DATE SEP 20 1994 <i>etc. 4</i> </div>	
9. Impact Level NA		

SEP 20 1994

ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT

600493

2. To: (Receiving Organization) Distribution	3. From: (Originating Organization) Process Laboratories & Technology	4. Related EDT No.: 600490
5. Proj./Prog./Dept./Div.: WM	6. Cog. Engr.: L. Jensen	7. Purchase Order No.: NA
8. Originator Remarks: Release of document "Statistical Characterization Report for Single-Shell Tank 241-T-111"		9. Equip./Component No.: NA
		10. System/Bldg./Facility: NA
11. Receiver Remarks:		12. Major Assm. Dwg. No.: NA
		13. Permit/Permit Application No.: NA
		14. Required Response Date: 9-30-94

15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	Impact Level	Reason for Transmittal	Originator Disposition	Receiver Disposition
1	WHC-SD-WM-TI-650		0	Statistical Characterization Report for Single-Shell Tank 241-T-111	NA	2	1	1

16. KEY			
Impact Level (F)	Reason for Transmittal (G)		Disposition (H) & (I)
1, 2, 3, or 4 (see MRP 5.43)	1. Approval 2. Release 3. Information	4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION (See Impact Level for required signatures)									
(G)	(H)	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN
Reason	Disp.								
2	1	Cog. Eng. L. Jensen	<i>[Signature]</i>	9/20/94	T6-07				
2	1	Cog. Mgr. J. P. Sloughier	<i>[Signature]</i>	9-20-94	T6-07				

18. Signature of EDT Originator <i>[Signature]</i> Date: 9/20/94	19. Authorized Representative Date for Receiving Organization Date: _____	20. Cognizant/Project Engineer's Manager <i>[Signature]</i> Date: 9-20-94	21. DOE APPROVAL (if required) Ltr. No. <input type="checkbox"/> Approved <input type="checkbox"/> Approved w/comments <input type="checkbox"/> Disapproved w/comments
---	---	--	--

BD-7400-172-2 (07/91) GEF097

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3.0 MEAN CONCENTRATION ESTIMATES	3-1
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3.2 STATISTICAL RESULTS	3-1
4.0 COMPARISON OF THE VARIANCE COMPONENT ESTIMATES	4-1
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4.2 STATISTICAL RESULTS	4-1
5.0 REFERENCES	5-1

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C MEAN CONCENTRATION CALCULATION METHODS	C-1

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LIST OF TERMS

222-S	Process and Analytical Laboratories
95% LL	lower limit of a 95% confidence interval on the mean
95% UL	upper limit of a 95% confidence interval on the mean
ANOVA	analysis of variance
T111	tank 241-T-111
CI	confidence interval
df	degrees of freedom
.dir	direct
DL	detection limit
GEA	gamma energy analysis
IC	ion chromatography
IC.w.	ion chromatography of a water leached sample
ICP	inductively coupled plasma analysis
ICP.a.	inductively coupled plasma analysis of an acid digested sample
ICP.f.	inductively coupled plasma analysis of a fusion digested sample
ICP.w.	inductively coupled plasma analysis of a water leached sample
.w.Spec	spectroscopic method of a water leached sample
KOH/Ni	Potassium Hydroxide/Nickel
Mean/DL	the mean of two sample results (sample and duplicate) divided by the detection limit
mL	milliliters
NA	not available
RAD.f.	radiochemistry analysis of a fusion digested sample
REML	restricted maximum likelihood estimation method
SST	single-shell tank
TGA	thermal gravimetric analysis
\bar{y}	weighted mean of the concentration data
$\sigma^2(\bar{y})$	estimated variance of \bar{y}
$\mu\text{Ci/L}$	microcuries per liter
$\mu\text{g/g}$	micrograms per gram
$\mu\text{Ci/g}$	microcuries per gram

STATISTICAL CHARACTERIZATION REPORT
FOR
SINGLE-SHELL TANK 241-T-111

1.0 SUMMARY

This report contains the results of the statistical analysis of data from two core samples obtained from Single-Shell Tank 241-T-111 (T111).

Section 2 contains a description of the core samples and the chemical analyses done on the core samples.

Section 3 contains mean concentration estimates and associated 95% confidence intervals (CI) on the mean for each of the analytes in T111.

Section 4 contains estimates of the spatial variability (variability between cores) and estimates of the "analytical error" from the core composite data.

Two types of analytical error were estimated from the core composite data: (1) compositing variability (variability between composite samples within the same core) and (2) analytical measurement error (variability between the primary and duplicate analyses within each core composite sample). Estimates of the analytical measurement error were used as the reference value to test the significance of the spatial and compositing variability. Spatial variability was significantly different than zero¹ for 39 out of 85 analytes in T111. The compositing variance was significantly different than zero for 39 out of the 85 analytes.

¹ Significantly different than zero at the $\alpha = 0.05$ level.

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2.0 INTRODUCTION

Two core samples (cores 31 and 33) were taken from T111. Cores 31 and 33 were obtained from risers 6 and 3, respectively. Figure 2-1 (p. 2-2) gives the locations of the risers. The segment recoveries for each core are given in Table 2-1. Core 31 had nearly 100% segment recovery for all segments except segments 1 and 6. There was nearly 100% segment recovery for all of the segments in Core 33.

Table 2-1. T-111 Core Recoveries.

Core	Segment Number								
	1	2	3	4	5	6	7	8	9
31	27%	80- 100%	95- 100%	80- 100%	100%	0%	90- 100%	100%	100%
33	100%	100%	87- 100%	75- 85%	88%	100%	100%	100%	100%

Due to the incomplete core sample recovery, the concentration estimates given in this report are biased. The magnitude of the bias is unknown.

Two core composite samples were made for each core sample from the homogenized solid segment waste. Primary and duplicate results were obtained from each core composite sample.

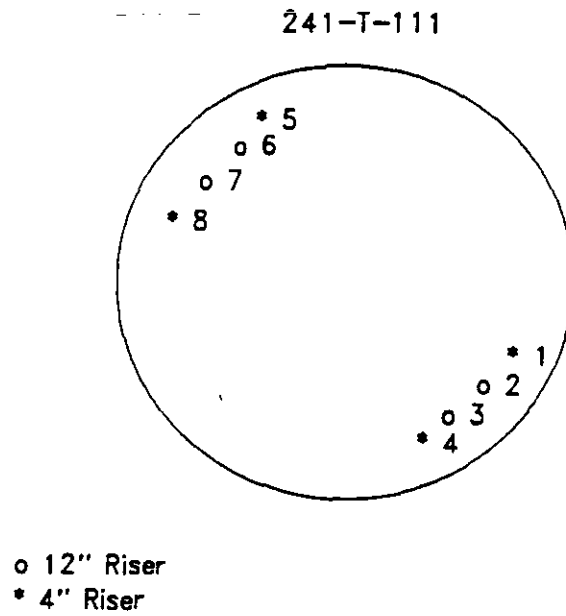
The chemical analysis of the primary and duplicate samples consisted of the following analytical methods and sample preparations (the notation for the method and preparation is also listed):

- inductively coupled plasma analysis (ICP) of an acid digested sample (ICP.a),
- ICP of a potassium hydroxide/nickel (KOH/Ni) fused sample, (ICP.f)
- ICP analysis of a water leached sample (ICP.w),
- ion chromatography (IC) of a water leached sample (IC.w),
- radio-chemistry, and
- gamma energy analysis (GEA).

Occasionally, special analytical methods are used on prepared and unprepared waste samples. The notation for the results from such methods is given in the tables of Appendix A and the tables in Sections 3 and 4.

Whenever possible, the results and data given here are identified by the analysis method, the type of dissolution and analyte. For example, the notation ICP.a.A1 refers to the A1 concentration from an ICP analysis of an acid digested sample.

Figure 2-1. Riser location.



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The core composite sample results are contained in Table A-1 (Appendix A). Ratios of the mean of each primary and duplicate result divided by the detection limit for that pair are also included. The ratios (Mean/DL) are provided to show how large the analyte concentrations are relative to the DL. The data packages for tank T111 (Kocher, 1993 and McKinney, 1993) contain a complete report of the sample results along with the laboratory quality control data. The core composite data for each analyte are illustrated in the figures provided in Appendix B.

Analysis of variance (ANOVA) models were fit to the data for all analytes that did not have any "less than values". All of the data available for statistical analysis, including the analytes with at least one "less than value," are reported in Table A-1. The ratios (Mean/DL) of all of the data are also reported in Table A-1.

From a quality of data perspective, it is desirable that the ANOVA models be applied to analyte concentrations greater than 10 times the detection limit. However, Analytical Evaluation and Reporting personnel, within the TWRS Information Management Systems, identified a list of "special analytes," given in Table 2-2. For this list, they requested that the ANOVA models be fit to the data provided the concentration values were greater than 3 times the detection limit or less than 10 times the detection limit.

Whenever possible the ANOVA models were fit using all of the data, regardless of the value of the detection limit. To warn the reader of potential detection limit problems the analytes in Tables 3-1, 4-1 and A-1 are denoted by a "•" or a "*" whenever a concentration is less than 3 times or 10 times the detection limit respectively. The results for such analytes are to be used with caution.

In Table A-1, some analytes are marked with a "*." Due to the dilution factors, the analytical result values are so close to the detection limit (DL), that the concentration values are questionable. For these analytes, ANOVA models were not fit to the data.

Table 2-2. Special Analyte List.

Aluminum	Nitrate
Bismuth	Nitrite
Calcium	Phosphate
Chromium	Carbonate
Iron	Fluoride
Silicon	Chloride
Sodium	Total Organic Carbon
Zirconium	Cyanide

A close examination of the figures in Appendix B reveals several "outlier data points" in the core composite data:

- ICP.a.Co The primary result for core 31, composite 2 of 11.7 $\mu\text{g/g}$ is over three times as large as the duplicate result. The other results for ICP.a.Co all fall in the range 2.7 to 3.8 $\mu\text{g/g}$. The detection limit for ICP.a.Co is 0.8 $\mu\text{g/g}$.

- ICP.a.Cu The duplicate result for core 31, composite 2 of 127 $\mu\text{g/g}$ is about four times as large as the primary result of 31.7 $\mu\text{g/g}$. The detection limit for ICP.a.Cu is 0.4 $\mu\text{g/g}$.
- NO_2 Both the primary and duplicate results from core 31, composite 2 are about one half the results for core 31, composite 1. The average of the results for core 31, composite 1, is 952 $\mu\text{g/g}$, the average for composite two is 525 $\mu\text{g/g}$. The detection limit for NO_2 (by water digestion spectrophotometric analysis) is 50 $\mu\text{g/g}$.

There is no direct evidence that the results noted above are due to analytical measurement errors. Consequently, the statistical analysis was performed on the data as it is reported in Table A-1. The potential anomalies in the data noted above can be clearly seen in the figures in Appendix B.

3.0 MEAN CONCENTRATION ESTIMATES

One of the tasks outlined in the Tank Waste Characterization Plan, Section 5.1.1.2 (Bell, 1993), is to estimate the constituent inventories in the waste. The inventories are estimated by computing mean concentrations and 95% confidence intervals (CIs) on the mean concentrations for each constituent. The estimate of the inventory and CI on the inventory of an analyte in the tank are equal to the corresponding mean concentration estimates and CI multiplied by the volume of waste in the tank. Therefore, estimates of tank inventory are not given in this document.

Table A-1 (Appendix A) contains the core composite data used to compute the mean concentration estimates and associated CIs.

3.1 STATISTICAL METHODS

The concentration estimates are given in the form of 95% CIs on the mean concentration. It is assumed that each primary sample and its duplicate are analyzed independently of one another. The two analytical results are used to estimate the analytical measurement error. Due to the hierarchical structure of the data, the analytical measurement error alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variance and the spatial variance is the appropriate variance of the mean for the CIs (Snedecor and Cochran, 1980). Appendix C contains a description of the statistical model and formulas used to calculate estimates of the mean, variance of the mean and the CI on the mean.

3.2 STATISTICAL RESULTS

Table 3-1 contains the summary statistics, by analyte, for ICP acid digestion, ICP water leach, ICP KOH/Ni fusion dissolution, radio chemistry and IC analyses. The summary statistics are defined as follows:

\bar{y}	mean of the concentration data,
$\hat{\sigma}^2(\bar{y})$	estimated variance of \bar{y} ,
df	degrees of freedom,
95% LL	lower limit to the 95% CI on the mean and
95% UL	upper limit to the 95% CI on the mean.

For some analytes the lower confidence limit (95% LL) was negative. Since concentrations are greater than or equal to zero, any negative 95% LL values were set equal to zero.

The confidence intervals in Table 3-1 are wide relative to the range of the data because only two cores were used to estimate the spatial variability. Two core samples is the minimum number of core samples needed to estimate a tank's spatial variability.

Table 3-1. Concentration Estimate Statistics (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$). (sheet 1 of 3)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
ICP.a.Ag	1.26E+02	7.86E+03	1	0.00	1.25E+03
ICP.a.Al	5.41E+02	1.06E+04	1	0.00	1.85E+03
ICP.a.B	2.80E+01	7.56E+00	1	0.00	6.30E+01
ICP.a.Ba	6.90E+01	6.46E+01	1	0.00	1.71E+02
ICP.a.Bi	2.59E+04	6.38E+06	1	0.00	5.80E+04
ICP.a.Ca	1.88E+03	2.12E+05	1	0.00	7.72E+03
ICP.a.Cd*	5.80E+00	3.03E+00	1	0.00	2.79E+01
ICP.a.Ce*	3.37E+01	9.69E+00	1	0.00	7.33E+01
ICP.a.Co*	4.30E+00	1.63E+00	1	0.00	2.05E+01
ICP.a.Cr	1.98E+03	1.63E+04	1	3.57E+02	3.60E+03
ICP.a.Cu	3.35E+01	3.53E+02	1	0.00	2.72E+02
ICP.a.Fe	1.85E+04	1.21E+06	1	4.55E+03	3.25E+04
ICP.a.K	1.14E+03	2.24E+03	1	5.34E+02	1.74E+03
ICP.a.La	4.22E+03	3.00E+05	1	0.00	1.12E+04
ICP.a.Mg	3.77E+02	6.36E+03	1	0.00	1.39E+03
ICP.a.Mn	6.33E+03	2.68E+04	1	4.25E+03	8.41E+03
ICP.a.Na	3.69E+04	1.56E+06	1	2.10E+04	5.27E+04
ICP.a.Ni	1.32E+02	5.12E+02	1	0.00	4.19E+02
ICP.a.P	1.03E+04	1.21E+05	1	5.90E+03	1.47E+04
ICP.a.Pb	3.47E+02	2.64E+04	1	0.00	2.41E+03
ICP.a.S	1.21E+03	1.06E+03	1	8.00E+02	1.63E+03
ICP.a.Sb*	3.14E+01	1.65E+01	1	0.00	8.30E+01
ICP.a.Si	4.69E+02	9.17E+02	1	8.40E+01	8.54E+02
ICP.a.Sr	3.00E+02	3.75E+02	1	5.39E+01	5.46E+02
ICP.a.Ti	1.95E+01	1.39E+02	1	0.00	1.69E+02
ICP.a.V	1.45E+01	6.58E+00	1	0.00	4.71E+01
ICP.a.Zn	6.50E+01	6.46E+02	1	0.00	3.88E+02
ICP.f.Ag	1.28E+02	8.05E+03	1	0.00	1.27E+03
ICP.f.Al	5.70E+02	9.70E+03	1	0.00	1.82E+03
ICP.f.Ba	6.46E+01	2.45E+01	1	1.73E+00	1.28E+02
ICP.f.Bi	2.36E+04	9.08E+06	1	0.00	6.18E+04
ICP.f.Ca*	2.42E+03	8.27E+04	1	0.00	6.07E+03
ICP.f.Cd*	8.12E+00	1.76E+00	1	0.00	2.50E+01
ICP.f.Ce*	1.15E+01	1.16E+00	1	0.00	2.52E+01
ICP.f.Cr	1.80E+03	1.56E+03	1	1.30E+03	2.30E+03

* : Analytes with a portion of the data below 3 times the DL.

* : Analytes with a portion of the data below 10 times the DL.

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Table 3-1. Concentration Estimate Statistics (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$). (sheet 2 of 3)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
ICP.f.Cu*	2.39E+01	3.56E+01	1	0.00	1.05E+02
ICP.f.Fe	1.80E+04	4.05E+06	1	0.00	4.36E+04
ICP.f.La	4.11E+03	3.08E+05	1	0.00	1.12E+04
ICP.f.Mg	3.55E+02	7.31E+03	1	0.00	1.44E+03
ICP.f.Mn	6.28E+03	1.88E+04	1	4.54E+03	8.02E+03
ICP.f.Na	3.70E+04	6.00E+06	1	5.82E+03	6.81E+04
ICP.f.Ni	8.14E+03	6.41E+06	1	0.00	4.03E+04
ICP.f.P	1.04E+04	8.42E+05	1	0.00	2.21E+04
ICP.f.Pb*	3.65E+02	9.38E+03	1	0.00	1.60E+03
ICP.f.S	1.23E+03	1.13E+04	1	0.00	2.58E+03
ICP.f.Si	5.67E+03	5.41E+04	1	2.71E+03	8.62E+03
ICP.f.Sr	2.98E+02	6.24E+01	1	1.97E+02	3.98E+02
ICP.f.Ti	4.79E+01	6.09E+02	1	0.00	3.62E+02
ICP.f.V*	1.47E+01	8.32E-01	1	3.06E+00	2.62E+01
ICP.f.Zn*	1.06E+02	7.17E+00	1	7.22E+01	1.40E+02
ICP.w.Al*	1.09E+01	5.75E+00	1	0.00	4.14E+01
ICP.w.B*	4.07E+00	6.77E-01	1	0.00	1.45E+01
ICP.w.Bi	2.02E+02	2.40E+03	1	0.00	8.24E+02
ICP.w.Ca*	6.16E+01	3.32E+01	1	0.00	1.35E+02
ICP.w.Cr	2.18E+02	2.45E+01	1	1.55E+02	2.81E+02
ICP.w.Fe	1.28E+02	3.11E+02	1	0.00	3.52E+02
ICP.w.K	7.19E+02	1.54E+03	1	2.21E+02	1.22E+03
ICP.w.La*	1.10E+01	1.42E+01	1	0.00	5.89E+01
ICP.w.Mg*	3.64E+00	5.45E-02	1	6.75E-01	6.61E+00
ICP.w.Mn	2.47E+01	2.36E+01	1	0.00	8.65E+01
ICP.w.Na	3.30E+04	2.44E+06	1	1.31E+04	5.28E+04
ICP.w.P	5.68E+03	3.24E+04	1	3.39E+03	7.97E+03
ICP.w.S	1.15E+03	2.38E+03	1	5.29E+02	1.77E+03
ICP.w.Si	5.72E+02	5.35E+03	1	0.00	1.50E+03
ICP.w.Sr*	1.96E+00	8.65E-02	1	0.00	5.70E+00
IC.w.Cl	4.50E+02	1.11E+03	1	2.56E+01	8.74E+02
IC.w.F	2.30E+03	6.46E+05	1	0.00	1.25E+04
IC.w.NO3	4.12E+04	7.77E+06	1	5.82E+03	7.67E+04
IC.w.PO4	1.55E+04	1.53E+06	1	0.00	3.13E+04
IC.w.SO4	3.54E+03	2.85E+04	1	1.40E+03	5.69E+03

*: Analytes with a portion of the data below 3 times the DL.
*: Analytes with a portion of the data below 10 times the DL.

Table 3-1. Concentration Estimate Statistics (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$). (sheet 3 of 3)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
GEA.Am-241	4.24E-02	2.61E-06	1	2.19E-02	6.29E-02
GEA.Cs-137	1.66E-01	3.35E-03	1	0.00	9.02E-01
Gross.alpha	3.73E-01	1.96E-04	1	1.95E-01	5.51E-01
Gross.beta	1.51E+01	3.48E+01	1	0.00	9.00E+01
TGA.X.H2O	7.65E+01	2.23E+01	1	1.64E+01	1.37E+02
Am-241*	4.26E-02	6.65E-25	1	4.26E-02	4.26E-02
Hg*	1.43E+00	1.53E-01	1	0.00	6.40E+00
NO2.W.Spec	7.93E+02	8.76E+03	1	0.00	1.98E+03
Percent.H2O	7.60E+01	5.81E-01	1	6.63E+01	8.57E+01
Pu-239/40	1.39E-01	9.19E-06	1	1.00E-01	1.77E-01
Sr-90	5.41E+00	3.53E+00	1	0.00	2.93E+01
TOC*	3.12E+03	3.83E+05	1	0.00	1.10E+04
Tc-99*	7.92E-03	8.90E-06	1	0.00	4.58E-02
U*	2.79E+03	2.01E+05	1	0.00	8.50E+03
pH	9.98E+00	7.79E-03	1	8.86E+00	1.11E+01

*: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

4.0 COMPARISON OF THE VARIANCE COMPONENT ESTIMATES

Using the hierarchical structure of the core composite data, estimates of the between core spatial variability ($\sigma^2(S)$), the compositing variability ($\sigma^2(C)$) and the analytical measurement variability ($\sigma^2(A)$) can be obtained. The spatial variance is a measure of the variability between cores. The compositing variance measures the variability between composite samples within the same core. The analytical measurement variance includes, among other things, the sample handling error and the chemical analysis error. This variance is a measure of the difference between the analytical results from the primary and duplicate samples.

The estimate of the variance of the mean is a linear function of the spatial, compositing and analytical measurement variances. To help evaluate the magnitude of these three variance components, estimates of each variance component are given in Table 4-1.

4.1 STATISTICAL METHODS

Estimates of the spatial variance ($\sigma^2(S)$), compositing variance ($\sigma^2(C)$) and analytical measurement variance ($\sigma^2(A)$), were obtained for each analyte using Restricted Maximum Likelihood Estimation (REML) methods. Restricted maximum likelihood estimation is discussed by Harville (1977).

To test the significance of the variance components, an analysis of variance (ANOVA) was calculated using the hierarchical statistical model described in Appendix C. The mean square error terms in the ANOVA tables were used to perform F-tests on the spatial variability and F-tests on the composite variability. These tests determine whether or not $\sigma^2(S)$ and $\sigma^2(C)$ are significantly different than zero. The p-values given in Table 4-1 were derived from the results of these tests.

4.2 STATISTICAL RESULTS

The REML estimates of each component of variability along with the p-values (significance level) from the F-tests are given in Table 4-1. A p-value less than 0.05 indicates that $\sigma^2(S)$ or $\sigma^2(C)$ is significantly different than zero at the 0.05 level.

The p-values from the tests on $\sigma^2(S)$ were less than 0.05 for 39 out of the 85 analytes in T111. This indicates that the differences between the two cores were statistically significant for these 39 cases.

The p-values from the tests on $\sigma^2(C)$ were less than 0.05 for 39 out of the 85 analytes in T111. This indicates that, relative to the analytical error, differences between composite samples were significantly different than zero in 39 cases. This means, for these 39 analytes, the two composite samples were statistically different from each other. Conversely, for 46 out of 85 cases, differences between composite samples were not statistically significant.

Table 4-1. Variance Component Estimates. (sheet 1 of 3)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
ICP.a.Ag	1.56E+04	0.001	1.94E+02	0.000	3.16E+00
ICP.a.Al	1.89E+04	0.055	4.79E+03	0.000	1.06E+01
ICP.a.B	1.25E+01	0.093	9.28E-01	0.387	8.59E+00
ICP.a.Ba	6.91E+01	0.263	1.20E+02	0.000	3.54E-01
ICP.a.Bi	1.27E+07	0.000	6.25E+03	0.360	3.75E+04
ICP.a.Ca	4.10E+05	0.010	2.17E+04	0.074	9.95E+03
ICP.a.Cd*	5.96E+00	0.004	1.90E-01	0.039	5.34E-02
ICP.a.Ce*	1.69E+01	0.064	9.66E-01	0.380	7.87E+00
ICP.a.Co*	8.92E-01	0.376	8.08E-01	0.389	7.84E+00
ICP.a.Cr	3.17E+04	0.007	1.36E+03	0.081	6.75E+02
ICP.a.Cu	3.39E+02	0.290	1.67E+02	0.369	1.14E+03
ICP.a.Fe	2.33E+06	0.013	1.55E+05	0.063	6.25E+04
ICP.a.K	2.45E-24	0.911	8.86E+03	0.000	1.75E+02
ICP.a.La	5.91E+05	0.003	1.63E+04	0.019	2.88E+03
ICP.a.Mg	1.25E+04	0.005	5.18E+02	0.001	1.58E+01
ICP.a.Mn	2.97E+04	0.254	4.29E+04	0.030	1.00E+04
ICP.a.Na	2.76E+06	0.057	6.73E+05	0.016	1.05E+05
ICP.a.Ni	1.02E+03	0.000	6.13E+00	0.091	3.38E+00
ICP.a.P	2.37E-22	0.608	4.77E+05	0.000	1.25E+04
ICP.a.Pb	5.20E+04	0.003	1.41E+03	0.000	1.91E+01
ICP.a.S	1.11E+03	0.268	1.94E+03	0.003	1.25E+02
ICP.a.Sb*	1.11E-20	0.739	1.44E-14	0.578	1.32E+02
ICP.a.Si	2.57E-31	0.946	7.61E-14	0.418	7.34E+03
ICP.a.Sr	6.38E+02	0.079	2.18E+02	0.004	1.54E+01
ICP.a.Ti	2.75E+02	0.001	4.69E+00	0.000	7.83E-02
ICP.a.V	1.83E+00	0.423	2.25E+01	0.000	2.30E-01
ICP.a.Zn	1.22E+03	0.021	1.35E+02	0.008	1.42E+01
ICP.f.Ag	1.61E+04	0.000	6.23E+00	0.295	1.83E+01
ICP.f.Al	1.90E+04	0.005	6.78E+02	0.030	1.57E+02
ICP.f.Ba	3.99E+01	0.102	1.71E+01	0.012	2.19E+00
ICP.f.Bi	1.81E+07	0.001	2.88E+04	0.392	2.94E+05
ICP.f.Ca*	1.51E+05	0.008	2.07E-19	0.804	5.86E+04
ICP.f.Cd*	2.17E+00	0.126	1.21E-20	0.649	5.38E+00
ICP.f.Co*	1.44E+00	0.078	1.01E-29	0.755	3.48E+00
ICP.f.Cr	3.72E-15	0.994	5.63E+03	0.013	1.21E+03

*: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

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Table 4-1. Variance Component Estimates. (sheet 2 of 3)

Analyte	$\sigma^2(s)$	Test: $\sigma^2(s)=0$ p-value	$\sigma^2(c)$	Test: $\sigma^2(c)=0$ p-value	$\sigma^2(A)$
ICP.f.Cu*	6.97E+01	0.005	2.00E+00	0.119	1.43E+00
ICP.f.Fe	8.00E+06	0.003	1.68E+05	0.079	8.13E+04
ICP.f.La	5.96E+05	0.010	3.82E+04	0.012	4.98E+03
ICP.f.Mg	1.46E+04	0.000	5.15E-44	0.913	1.60E+02
ICP.f.Mn	1.66E-11	0.553	7.02E+04	0.011	9.70E+03
ICP.f.Na	1.17E+07	0.007	5.43E+05	0.044	1.65E+05
ICP.f.Ni	1.17E+07	0.033	4.18E-12	0.498	4.39E+06
ICP.f.P	1.57E+06	0.028	1.95E+05	0.049	6.45E+04
ICP.f.Pb*	1.85E+04	0.003	4.35E+02	0.030	1.02E+02
ICP.f.S	2.15E+04	0.017	2.00E+03	0.008	2.13E+02
ICP.f.Si	1.06E+05	0.005	1.00E+03	0.369	6.83E+03
ICP.f.Sr	6.12E-16	0.661	1.15E+02	0.222	2.69E+02
ICP.f.Ti	1.22E+03	0.000	2.66E-01	0.331	1.12E+00
ICP.f.V*	5.86E-19	0.905	2.17E+00	0.107	2.31E+00
ICP.f.Zn*	5.69E-23	0.440	1.10E-22	0.839	5.74E+01
ICP.w.Al*	7.35E+00	0.208	6.93E+00	0.061	2.72E+00
ICP.w.B*	1.14E+00	0.082	3.99E-01	0.008	3.99E-02
ICP.w.Bi	3.90E+03	0.102	1.30E+03	0.123	9.66E+02
ICP.w.Ca*	1.86E-11	0.144	1.69E-22	0.862	2.66E+02
ICP.w.Cr	5.66E-20	0.979	9.52E+01	0.001	5.62E+00
ICP.w.Fe	6.16E+01	0.436	9.05E+02	0.077	4.29E+02
ICP.w.K	2.28E+03	0.148	1.57E+03	0.001	4.73E+01
ICP.w.La*	2.71E+01	0.015	1.34E+00	0.226	2.23E+00
ICP.w.Mg*	3.04E-21	0.597	1.61E-01	0.098	1.14E-01
ICP.w.Mn	2.51E+01	0.266	3.19E+01	0.130	2.51E+01
ICP.w.Na	4.53E+06	0.030	6.84E+05	0.002	2.88E+04
ICP.w.P	3.98E+04	0.222	3.94E+04	0.089	2.13E+04
ICP.w.S	3.95E+03	0.091	1.50E+03	0.014	2.13E+02
ICP.w.Si	8.53E+03	0.113	1.98E+03	0.272	4.72E+03
ICP.w.Sr*	6.35E-02	0.340	1.83E-01	0.061	7.19E-02
IC.w.Cl	1.99E+03	0.043	1.15E-10	0.509	9.66E+02
IC.w.F	1.28E+06	0.001	1.37E+04	0.102	8.44E+03
IC.w.NO3	1.42E+07	0.038	2.46E+06	0.013	3.44E+05
IC.w.PO4	2.59E+06	0.082	8.54E+05	0.030	1.99E+05

*: Analytes with a portion of the data below 3 times the DL.
*: Analytes with a portion of the data below 10 times the DL.

Table 4-1. Variance Component Estimates. (sheet 3 of 3)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
IC.W.S04	5.27E+04	0.032	7.04E+03	0.071	3.14E+03
GEA-Am-241	2.99E-32	0.786	8.13E-06	0.062	4.59E-06
GEA.Cs-137	6.61E-03	0.003	1.87E-04	0.000	1.88E-06
Gross.alpha	3.42E-04	0.065	7.74E-05	0.098	4.58E-05
Gross.beta	6.93E+01	0.000	3.14E-01	0.026	6.62E-02
TGA.X.H2O	3.17E+01	0.008	3.78E-33	0.935	5.18E+01
Am-241*	6.65E-25	0.976	7.41E-06	0.173	1.66E-05
Hg*	1.53E-01	0.022	1.36E-02	0.115	9.38E-03
NO2.W.Spec	1.92E-14	0.789	3.46E+04	0.000	9.50E+02
Percent.H2O	8.99E-01	0.128	2.55E-01	0.259	5.46E-01
Pu-239/40	2.49E-28	0.736	2.01E-05	0.171	3.33E-05
Sr-90	7.04E+00	0.000	1.79E-02	0.209	2.61E-02
TOC*	6.10E+05	0.113	2.98E+05	0.006	2.72E+04
Tc-99*	1.76E-05	0.001	2.63E-07	0.027	5.75E-08
U*	6.24E-06	0.831	7.91E+05	0.001	2.99E+04
pH	7.11E-28	0.601	3.06E-02	0.001	1.19E-03

*: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

5.0 REFERENCES

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APPENDIX A

CORE COMPOSITE DATA

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Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 1 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.a.Ag	2.02E+02 2.03E+02	4.05E+02	2.25E+02 2.28E+02	4.53E+02	4.48E+01 4.39E+01	8.87E+01	2.81E+01 3.19E+01	6.00E+01
ICP.a.Al	5.80E+02 5.88E+02	2.43E+02	7.03E+02 7.07E+02	2.94E+02	4.71E+02 4.73E+02	1.97E+02	4.05E+02 4.04E+02	1.69E+02
ICP.a.As	*3.13E+00 3.17E+00	NA	*3.02E+00 *2.81E+00	NA	3.39E+00 3.50E+00	1.15E+00	3.23E+00 *2.90E+00	NA
ICP.a.B	2.34E+01 3.08E+01	4.52E+01	2.53E+01 2.16E+01	3.91E+01	2.92E+01 2.96E+01	4.90E+01	3.20E+01 3.23E+01	5.36E+01
ICP.a.Ba	5.66E+01 5.75E+01	1.90E+02	6.53E+01 6.46E+01	2.17E+02	6.67E+01 6.70E+01	2.23E+02	8.67E+01 8.79E+01	2.91E+02
ICP.a.Be	*1.04E-01 1.06E-01	NA	*1.01E-01 *9.38E-02	NA	1.13E-01 1.20E-01	1.17E+00	1.08E-01 *1.00E-01	NA
ICP.a.Bi	2.34E+04 2.37E+04	3.14E+03	2.33E+04 2.32E+04	3.10E+03	2.84E+04 2.86E+04	3.80E+03	2.86E+04 2.82E+04	3.79E+03
ICP.a.Ca	2.13E+03 2.26E+03	4.99E+02	2.36E+03 2.61E+03	5.65E+02	1.49E+03 1.50E+03	3.40E+02	1.35E+03 1.34E+03	3.06E+02
ICP.a.Cd*	7.19E+00 7.25E+00	1.81E+01	7.78E+00 7.94E+00	1.97E+01	4.09E+00 4.70E+00	1.10E+01	3.64E+00 3.80E+00	9.30E+00
ICP.a.Ce*	3.39E+01 3.13E+01	3.23E+00	2.90E+01 2.83E+01	2.84E+00	4.00E+01 3.57E+01	3.75E+00	3.28E+01 3.89E+01	3.55E+00
ICP.a.Co*	3.42E+00 3.38E+00	4.25E+00	3.79E+00 1.17E+01	9.68E+00	3.15E+00 3.10E+00	3.91E+00	3.13E+00 2.70E+00	3.64E+00
ICP.a.Cr	1.83E+03 1.89E+03	2.07E+03	1.84E+03 1.84E+03	2.04E+03	2.05E+03 2.08E+03	2.29E+03	2.16E+03 2.13E+03	2.38E+03
ICP.a.Cu	2.48E+01 2.57E+01	6.31E+01	1.27E+02 3.17E+01	1.98E+02	1.64E+01 1.65E+01	4.11E+01	1.29E+01 1.30E+01	3.24E+01
ICP.a.Fe	1.89E+04 1.95E+04	1.92E+04	2.01E+04 2.00E+04	2.01E+04	1.74E+04 1.76E+04	1.75E+04	1.75E+04 1.72E+04	1.74E+04
ICP.a.K	1.08E+03 1.11E+03	9.78E+01	1.22E+03 1.20E+03	1.08E+02	1.21E+03 1.22E+03	1.08E+02	1.02E+03 1.02E+03	9.11E+01

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

: Analytes with a portion of the data below 3 times the DL.

* : Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 2 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.a.La	3.66E+03 3.78E+03	2.66E+03	3.64E+03 3.59E+03	2.58E+03	4.61E+03 4.67E+03	3.31E+03	4.91E+03 4.86E+03	3.49E+03
ICP.a.Mg	4.32E+02 4.38E+02	1.45E+03	4.75E+02 4.82E+02	1.60E+03	3.02E+02 3.07E+02	1.02E+03	2.88E+02 2.92E+02	9.67E+02
ICP.a.Mn	6.07E+03 6.31E+03	3.10E+04	6.14E+03 6.14E+03	3.07E+04	6.65E+03 6.77E+03	3.36E+04	6.32E+03 6.23E+03	3.14E+04
ICP.a.Na	3.71E+04 3.80E+04	1.21E+04	3.87E+04 3.86E+04	1.25E+04	3.49E+04 3.50E+04	1.13E+04	3.63E+04 3.62E+04	1.17E+04
ICP.a.Ni	1.49E+02 1.54E+02	8.91E+01	1.57E+02 1.57E+02	9.24E+01	1.09E+02 1.10E+02	6.44E+01	1.09E+02 1.08E+02	6.38E+01
ICP.a.P	1.02E+04 1.00E+04	1.74E+03	9.94E+03 9.98E+03	1.72E+03	9.97E+03 9.75E+03	1.70E+03	1.13E+04 1.14E+04	1.96E+03
ICP.a.Pb	4.69E+02 4.81E+02	7.66E+01	5.42E+02 5.44E+02	8.76E+01	2.00E+02 2.01E+02	3.23E+01	1.67E+02 1.69E+02	2.71E+01
ICP.a.S	1.21E+03 1.24E+03	4.54E+02	1.26E+03 1.27E+03	4.69E+02	1.14E+03 1.14E+03	4.22E+02	1.22E+03 1.22E+03	4.52E+02
ICP.a.Sb*	3.90E+01 2.19E+01	1.72E+00	3.61E+01 3.69E+01	2.06E+00	2.00E+01 5.18E+01	2.03E+00	1.91E+01 2.61E+01	1.28E+00
ICP.a.Se	*7.92E+00 8.04E+00	NA	*7.66E+00 *7.13E+00	NA	8.60E+00 1.20E+01	1.36E+00	8.18E+00 *7.40E+00	NA
ICP.a.Si	5.29E+02 4.36E+02	3.71E+02	4.18E+02 5.24E+02	3.62E+02	5.75E+02 4.80E+02	4.06E+02	4.90E+02 2.98E+02	3.03E+02
ICP.a.Sn	4.28E+00 4.13E+00	2.63E+00	1.74E+00 3.13E+00	1.52E+00	1.81E+00 1.80E+00	1.13E+00	1.72E+00 *1.50E+00	NA
ICP.a.Sr	2.78E+02 2.85E+02	9.38E+02	2.80E+02 2.80E+02	9.33E+02	3.01E+02 3.08E+02	1.02E+03	3.37E+02 3.32E+02	1.12E+03
ICP.a.Ti	2.96E+01 2.93E+01	7.36E+01	3.34E+01 3.27E+01	8.26E+01	9.00E+00 8.80E+00	2.23E+01	6.42E+00 6.50E+00	1.62E+01
ICP.a.V	1.23E+01 1.31E+01	2.54E+01	2.13E+01 2.15E+01	4.28E+01	1.35E+01 1.42E+01	2.77E+01	9.58E+00 1.04E+01	2.00E+01

NA: Not Available.

*: Value less than DL or less than 5 percent above the DL.

•: Analytes with a portion of the data below 3 times the DL.

+: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 3 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.a.Zn	7.68E+01 8.20E+01	2.65E+02	9.68E+01 1.06E+02	3.38E+02	4.36E+01 4.47E+01	1.47E+02	3.45E+01 3.54E+01	1.17E+02
ICP.a.Zr	8.34E-01 8.47E-01	NA	8.07E-01 7.51E-01	NA	9.05E-01 9.20E-01	NA	8.61E-01 7.70E-01	NA
ICP.f.Ag	2.10E+02 2.18E+02	8.56E+01	2.17E+02 2.26E+02	8.86E+01	3.89E+01 4.00E+01	1.36E+01	3.69E+01 3.74E+01	1.28E+01
ICP.f.Al	6.32E+02 6.56E+02	5.37E+01	6.80E+02 7.06E+02	5.78E+01	4.83E+02 4.85E+02	4.03E+01	4.59E+02 4.59E+02	3.83E+01
ICP.f.As	*1.50E+01 *1.50E+01	NA	*1.50E+01 *1.50E+01	NA	*1.50E+01 *1.50E+01	NA	*1.50E+02 *1.50E+02	NA
ICP.f.B	*3.01E+00 *3.00E+00	NA	*2.99E+00 *2.99E+00	NA	*5.36E+00 *4.32E+00	NA	*4.58E+00 *5.10E+00	NA
ICP.f.Ba	5.72E+01 6.04E+01	3.92E+01	5.96E+01 6.15E+01	4.04E+01	6.60E+01 6.48E+01	4.36E+01	7.45E+01 7.30E+01	4.92E+01
ICP.f.Be	*5.01E-01 *5.00E-01	NA	*4.99E-01 *4.99E-01	NA	*4.99E-01 *5.00E-01	NA	*5.00E-01 *4.99E-01	NA
ICP.f.Bi	2.05E+04 2.14E+04	5.59E+02	2.02E+04 2.01E+04	5.37E+02	2.66E+04 2.63E+04	7.05E+02	2.73E+04 2.61E+04	7.12E+02
ICP.f.Ca*	2.58E+03 2.93E+03	1.25E+02	2.49E+03 2.83E+03	1.21E+02	2.50E+03 1.93E+03	9.23E+00	2.18E+03 1.92E+03	8.54E+00
ICP.f.Cd*	7.34E+00 9.16E+00	4.13E+00	7.18E+00 1.41E+01	5.32E+00	6.76E+00 6.08E+00	3.21E+00	6.86E+00 7.48E+00	3.59E+00
ICP.f.Ce	*5.06E+01 *5.05E+01	NA	*5.04E+01 *5.04E+01	NA	*5.04E+01 *5.05E+01	NA	*5.05E+01 *5.04E+01	NA
ICP.f.Co*	9.70E+00 1.05E+01	2.53E+00	1.11E+01 1.05E+01	2.70E+00	1.37E+01 1.30E+01	3.34E+00	8.90E+00 1.48E+01	2.96E+00
ICP.f.Cr	1.86E+03 1.92E+03	4.20E+02	1.67E+03 1.73E+03	3.78E+02	1.81E+03 1.76E+03	3.97E+02	1.82E+03 1.82E+03	4.04E+02
ICP.f.Cu*	3.68E+01 3.59E+01	1.82E+01	3.41E+01 3.42E+01	1.71E+01	2.16E+01 2.26E+01	9.82E+00	2.30E+01 2.61E+01	1.09E+01

NA: Not Available.

*: Value less than DL or less than 5 percent above the DL.

.: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 4 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.f.Fe	2.02E+04 2.08E+04	4.10E+03	1.95E+04 1.97E+04	3.92E+03	1.62E+04 1.57E+04	6.16E+02	1.61E+04 1.61E+04	6.22E+02
ICP.f.K	*5.61E+01 *5.60E+01	NA	*5.59E+01 *5.59E+01	NA	*5.59E+01 *5.60E+01	NA	*5.60E+01 *5.59E+01	NA
ICP.f.La	3.63E+03 3.75E+03	5.27E+02	3.38E+03 3.45E+03	4.88E+02	4.58E+03 4.45E+03	6.45E+02	4.84E+03 4.78E+03	6.87E+02
ICP.f.Mg	4.24E+02 4.52E+02	2.92E+02	4.31E+02 4.56E+02	2.96E+02	2.78E+02 2.58E+02	3.46E+01	2.74E+02 2.69E+02	3.50E+01
ICP.f.Mn	6.29E+03 6.47E+03	6.38E+03	5.86E+03 6.02E+03	5.94E+03	6.29E+03 6.15E+03	4.01E+02	6.59E+03 6.59E+03	4.25E+02
ICP.f.Na	3.96E+04 4.01E+04	2.57E+03	3.85E+04 3.94E+04	2.51E+03	3.41E+04 3.36E+04	5.34E+01	3.51E+04 3.52E+04	5.54E+01
ICP.f.Ni	5.94E+03 5.61E+03	6.79E+02	7.49E+03 3.40E+03	6.41E+02	9.49E+03 9.09E+03	8.68E+01	9.81E+03 1.43E+04	1.13E+02
ICP.f.P	1.12E+04 1.19E+04	3.98E+02	1.11E+04 1.11E+04	3.83E+02	8.99E+03 9.15E+03	1.04E+02	9.91E+03 9.91E+03	1.13E+02
ICP.f.Pb+	4.27E+02 4.53E+02	1.42E+01	4.82E+02 4.86E+02	1.56E+01	2.62E+02 2.72E+02	8.61E+00	2.72E+02 2.67E+02	8.69E+00
ICP.f.S	1.35E+03 1.36E+03	1.00E+02	1.29E+03 1.33E+03	9.70E+01	1.08E+03 1.08E+03	8.00E+01	1.16E+03 1.16E+03	8.59E+01
ICP.f.Sb	*8.87E+01 *8.85E+01	NA	*8.83E+01 *8.83E+01	NA	1.29E+02 *8.85E+01	NA	*8.85E+01 *8.83E+01	NA
ICP.f.Se	*3.81E+01 *3.80E+01	NA	*3.79E+01 *3.79E+01	NA	*3.79E+01 *3.80E+01	NA	*3.80E+01 *3.79E+01	NA
ICP.f.Si	5.88E+03 6.04E+03	9.17E+02	5.78E+03 5.89E+03	8.98E+02	5.52E+03 5.39E+03	7.43E+01	5.41E+03 5.41E+03	7.37E+01
ICP.f.Sn	*8.02E+00 *8.00E+00	NA	*7.98E+00 *7.98E+00	NA	*7.98E+00 *8.00E+00	NA	*8.00E+00 *7.98E+00	NA
ICP.f.Sr	3.08E+02 2.98E+02	2.02E+02	2.70E+02 2.91E+02	1.87E+02	2.97E+02 2.84E+02	6.65E+01	2.98E+02 3.36E+02	7.25E+01

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

• : Analytes with a portion of the data below 3 times the DL.

* : Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 5 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.f.Tl	7.32E+01 7.25E+01	3.64E+01	7.13E+01 7.34E+01	3.62E+01	2.25E+01 2.22E+01	1.12E+01	2.51E+01 2.31E+01	1.21E+01
ICP.f.Va	1.11E+01 1.32E+01	4.86E+00	1.55E+01 1.74E+01	6.58E+00	1.38E+01 1.69E+01	6.14E+00	1.42E+01 1.51E+01	5.86E+00
ICP.f.Zn*	9.71E+01 1.11E+02	6.94E+01	1.11E+02 1.00E+02	7.03E+01	1.10E+02 1.00E+02	7.66E+00	1.02E+02 1.19E+02	8.07E+00
ICP.f.Zr	4.01E+00 4.00E+00	NA	3.99E+00 3.99E+00	NA	*3.99E+00 *4.00E+00	NA	*4.00E+00 *3.99E+00	NA
ICP.w.Ag	7.80E-01 *5.00E-01	NA	1.26E+00 1.07E+00	2.33E+00	*4.99E-01 6.08E-01	NA	*4.99E-01 *5.00E-01	NA
ICP.w.Al*	6.99E+00 6.43E+00	2.80E+00	1.04E+01 1.02E+01	4.29E+00	1.34E+01 1.77E+01	6.48E+00	1.19E+01 1.02E+01	4.60E+00
ICP.w.As	*3.00E+00 *3.00E+00	NA	*3.00E+00 *3.00E+00	NA	*2.99E+00 *2.99E+00	NA	*2.99E+00 *3.00E+00	NA
ICP.w.B*	3.50E+00 3.11E+00	5.51E+00	3.27E+00 3.12E+00	5.33E+00	5.54E+00 5.54E+00	9.23E+00	4.44E+00 4.06E+00	7.08E+00
ICP.w.Ba	*3.09E-01 *3.00E-01	NA	*3.00E-01 5.32E-01	NA	3.78E-01 7.15E-01	1.82E+00	5.32E-01 5.01E-01	1.72E+00
ICP.w.Be	*1.00E-01 *9.99E-02	NA	*9.99E-02 *9.99E-02	NA	*9.98E-02 *9.98E-02	NA	*9.98E-02 *9.99E-02	NA
ICP.w.Bi	8.34E+01 1.47E+02	1.54E+01	1.76E+02 2.05E+02	2.54E+01	2.05E+02 2.58E+02	3.09E+01	2.73E+02 2.67E+02	3.60E+01
ICP.w.Ca*	5.05E+01 5.12E+01	1.16E+01	5.43E+01 6.89E+01	1.40E+01	7.16E+01 6.13E+01	1.51E+01	9.35E+01 4.15E+01	1.53E+01
ICP.w.Cd	*4.00E-01 *4.00E-01	NA	*4.00E-01 *4.00E-01	NA	*3.99E-01 *3.99E-01	NA	*3.99E-01 *4.00E-01	NA
ICP.w.Ce	*1.01E+01 *1.01E+01	NA	*1.01E+01 *1.01E+01	NA	*1.01E+01 *1.01E+01	NA	*1.01E+01 *1.01E+01	NA
ICP.w.Co	*8.00E-01 *7.99E-01	NA	*7.99E-01 8.51E-01	NA	*7.98E-01 8.43E-01	NA	8.50E-01 *7.99E-01	NA

NA: Not Available.

*: Value less than DL or less than 5 percent above the DL.

.: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 6 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.W.Cr	2.11E+02 2.07E+02	2.32E+02	2.28E+02 2.30E+02	2.54E+02	2.22E+02 2.26E+02	2.49E+02	2.12E+02 2.09E+02	2.34E+02
ICP.W.Cu	*4.00E-01 *4.00E-01	NA	*4.00E-01 *4.00E-01	NA	*3.99E-01 *3.99E-01	NA	*3.99E-01 *4.00E-01	NA
ICP.W.Fe	5.70E+01 1.02E+02	7.95E+01	1.30E+02 1.51E+02	1.41E+02	1.16E+02 1.47E+02	1.32E+02	1.60E+02 1.58E+02	1.59E+02
ICP.W.K	7.40E+02 7.28E+02	6.55E+01	7.83E+02 7.83E+02	6.99E+01	7.04E+02 7.19E+02	6.35E+01	6.47E+02 6.50E+02	5.79E+01
ICP.W.La+	5.00E+00 7.05E+00	4.30E+00	7.69E+00 9.35E+00	6.09E+00	1.22E+01 1.55E+01	9.89E+00	1.57E+01 1.58E+01	1.13E+01
ICP.W.Mg+	2.67E+00 3.22E+00	9.82E+00	3.66E+00 4.23E+00	1.32E+01	3.60E+00 4.08E+00	1.28E+01	3.95E+00 3.72E+00	1.28E+01
ICP.W.Mn	1.01E+01 1.93E+01	7.35E+01	2.31E+01 2.70E+01	1.25E+02	2.04E+01 3.04E+01	1.27E+02	3.34E+01 3.42E+01	1.69E+02
ICP.W.Na	3.41E+04 3.39E+04	1.10E+04	3.51E+04 3.50E+04	1.13E+04	3.06E+04 3.09E+04	9.92E+03	3.19E+04 3.22E+04	1.03E+04
ICP.W.Ni	*1.70E+00 *1.70E+00	NA	*1.70E+00 *1.70E+00	NA	*1.70E+00 *1.70E+00	NA	*1.70E+00 *1.70E+00	NA
ICP.W.P	5.63E+03 5.89E+03	9.93E+02	5.81E+03 6.11E+03	1.03E+03	5.26E+03 5.34E+03	9.14E+02	5.66E+03 5.74E+03	9.83E+02
ICP.W.Pb	7.91E+00 *6.19E+00	NA	6.93E+00 8.92E+00	1.28E+00	*6.19E+00 *6.28E+00	NA	*6.19E+00 *6.19E+00	NA
ICP.W.S	1.21E+03 1.18E+03	4.43E+02	1.19E+03 1.21E+03	4.44E+02	1.05E+03 1.07E+03	3.93E+02	1.14E+03 1.14E+03	4.22E+02
ICP.W.Sb	*1.77E+01 *1.77E+01	NA	*1.77E+01 *1.77E+01	NA	*1.77E+01 *1.77E+01	NA	*1.77E+01 *1.77E+01	NA
ICP.W.Se	*7.60E+00 *7.59E+00	NA	*7.59E+00 8.44E+00	NA	*7.58E+00 *7.58E+00	NA	*7.58E+00 *7.59E+00	NA
ICP.W.Si	3.45E+02 5.30E+02	3.37E+02	5.30E+02 5.89E+02	4.30E+02	6.68E+02 6.71E+02	5.15E+02	6.22E+02 6.18E+02	4.77E+02

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

• : Analytes with a portion of the data below 3 times the DL.

* : Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 7 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
ICP.W.Sn	*1.60E+00 *1.60E+00	NA	*1.60E+00 *1.60E+00	NA	*1.60E+00 *1.60E+00	NA	*1.60E+00 *1.60E+00	NA
ICP.W.Sr+	9.37E-01 1.48E+00	4.03E+00	1.97E+00 2.29E+00	7.10E+00	1.97E+00 2.38E+00	7.25E+00	2.39E+00 2.29E+00	7.80E+00
ICP.W.Ti	*4.00E-01 *4.00E-01	NA	*4.00E-01 *4.00E-01	NA	*3.99E-01 *3.99E-01	NA	*3.99E-01 *4.00E-01	NA
ICP.W.V	*5.00E-01 *5.00E-01	NA	5.94E-01 8.20E-01	1.41E+00	*4.99E-01 *4.99E-01	NA	9.21E-01 6.76E-01	1.60E+00
ICP.W.Zn	*3.00E-01 *3.00E-01	NA	*3.00E-01 *3.00E-01	NA	*2.99E-01 *2.99E-01	NA	*2.99E-01 *3.00E-01	NA
ICP.W.Zr	8.00E-01 7.99E-01	NA	7.99E-01 7.99E-01	NA	7.98E-01 7.98E-01	NA	7.98E-01 7.99E-01	NA
IC.W.Cl	4.73E+02 4.66E+02	4.70E+01	5.18E+02 4.75E+02	4.97E+01	4.40E+02 3.62E+02	4.01E+01	4.40E+02 4.23E+02	4.32E+01
IC.W.F	3.14E+03 3.03E+03	3.09E+02	3.16E+03 3.09E+03	3.13E+02	1.47E+03 1.26E+03	1.37E+02	1.67E+03 1.59E+03	1.63E+02
IC.W.NO2	<1.10E+03 <1.10E+03	NA	8.71E+02 <1.10E+03	NA	8.42E+02 7.04E+02	7.73E+00	7.59E+02 7.04E+02	7.32E+00
IC.W.NO3	4.45E+04 4.41E+04	4.43E+02	4.36E+04 4.39E+04	4.38E+02	3.76E+04 3.61E+04	3.69E+02	3.98E+04 4.03E+04	4.01E+02
IC.W.PO4	1.56E+04 1.67E+04	1.62E+02	1.71E+04 1.77E+04	1.74E+02	1.36E+04 1.35E+04	1.36E+02	1.51E+04 1.50E+04	1.51E+02
IC.W.SO4	3.69E+03 3.69E+03	3.69E+01	3.72E+03 3.75E+03	3.74E+01	3.23E+03 3.34E+03	3.29E+01	3.41E+03 3.52E+03	3.47E+01
GEA.Am-241	4.37E-02 4.80E-02	4.02E+01	4.02E-02 4.16E-02	3.59E+01	3.95E-02 3.79E-02	3.39E+01	4.24E-02 4.61E-02	3.88E+02
GEA.Co-60	<4.21E-04 <3.39E-04	NA	<3.85E-04 <3.75E-04	NA	<3.45E-04 <3.70E-04	NA	<3.29E-04 <3.45E-04	NA
GEA.Cs-137	2.12E-01 2.11E-01	5.72E+02	2.36E-01 2.38E-01	6.41E+02	1.15E-01 1.12E-01	3.07E+02	1.03E-01 1.04E-01	2.80E+02

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

.: Analytes with a portion of the data below 3 times the DL.

*: Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 8 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
GEA.Eu-154	*1.09E-03 *1.07E-03	NA	<1.06E-03 3.24E-03	NA	<1.11E-03 *1.01E-03	NA	<1.05E-03 *9.64E-04	NA
GEA.Eu-155	<2.08E-03 *2.09E-03	NA	<2.13E-03 *2.12E-03	NA	2.97E-03 3.16E-03	5.15E+00	<1.49E-03 *1.49E-03	NA
Gross.alpha	3.59E-01 3.58E-01	5.05E+01	3.50E-01 3.69E-01	5.06E+01	3.78E-01 3.76E-01	7.11E+02	3.97E-01 3.97E-01	7.49E+02
Gross.beta	2.04E+01 2.07E+01	2.20E+02	2.16E+01 2.13E+01	2.29E+02	9.86E+00 9.32E+00	1.56E+02	8.95E+00 8.71E+00	1.44E+02
TIC.w.CO3	<5.00E+02 6.50E+02	NA	6.49E+02 9.99E+02	1.65E+00	7.49E+02 8.98E+02	1.65E+00	7.99E+02 1.40E+03	2.20E+00
TGA.X.H2O	6.13E+01 8.53E+01	NA	6.92E+01 7.12E+01	NA	8.10E+01 8.22E+01	NA	7.86E+01 8.30E+01	NA
Am-241*	4.43E-02 3.85E-02	7.26E+00	4.66E-02 3.96E-02	7.56E+00	3.71E-02 3.94E-02	6.59E+00	4.44E-02 3.11E-02	7.96E+00
C-14	3.80E-04 *2.25E-04	NA	<2.23E-04 *2.25E-04	NA	<2.25E-04 *2.25E-04	NA	<2.25E-04 *2.25E-04	NA
CN	<4.50E+00 *4.00E+00	NA	<3.41E+00 *3.57E+00	NA	<4.90E+00 *4.81E+00	NA	<4.76E+00 *4.61E+00	NA
H3	<3.15E-04 *3.15E-04	NA	<3.12E-04 *3.15E-04	NA	<3.15E-04 *3.15E-04	NA	<3.15E-04 *3.15E-04	NA
Hg*	1.70E+00 1.48E+00	1.27E+01	1.79E+00 1.88E+00	1.47E+01	1.18E+00 1.22E+00	9.60E+00	1.02E+00 1.15E+00	8.68E+00
I-129	<1.74E-02 *1.72E-02	NA	<2.40E-02 *1.92E-02	NA	<2.15E-02 *1.44E-02	NA	<1.75E-02 *2.17E-02	NA
NH3	<4.50E+03 *4.50E+03	NA	<4.50E+03 *4.50E+03	NA	<4.50E+03 *4.50E+03	NA	<4.50E+03 *4.50E+03	NA
NO2.w.Spec	9.49E+02 9.55E+02	1.90E+01	5.22E+02 5.27E+02	1.05E+01	8.72E+02 8.84E+02	1.76E+01	7.74E+02 8.60E+02	1.63E+01
Np-237	<3.25E-02 *3.24E-02	NA	<3.24E-02 *3.24E-02	NA	<3.24E-02 *3.24E-02	NA	<3.24E-02 *3.24E-02	NA

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

• : Analytes with a portion of the data below 3 times the DL.

* : Analytes with a portion of the data below 10 times the DL.

Table A-1. Core Composite Data (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$). (sheet 9 of 9)

Analyte	Core 31 Comp. 1	Mean/DL	Core 31 Comp. 2	Mean/DL	Core 33 Comp. 1	Mean/DL	Core 33 Comp. 2	Mean/DL
Percent. H ₂ O	7.48E+01 7.44E+01	NA	7.59E+01 7.59E+01	NA	7.72E+01 7.57E+01	NA	7.78E+01 7.64E+01	NA
Pu-238	<1.04E-02 <1.07E-02	NA	<1.03E-02 <1.04E-02	NA	<1.05E-02 <1.00E-02	NA	<1.13E-02 <1.04E-02	NA
Pu-239/40	1.35E-01 1.41E-01	3.83E+01	1.34E-01 1.37E-01	3.76E+01	1.39E-01 1.29E-01	3.83E+01	1.53E-01 1.62E-01	4.21E+01
Se	<6.05E-05 <6.11E-05	NA	6.17E-05 6.31E-05	1.11E+00	<1.23E-04 <1.26E-04	NA	<1.29E-04 <1.26E-04	NA
Sr-90	6.97E+00 7.34E+00	1.01E+03	7.55E+00 7.31E+00	1.62E+03	3.67E+00 3.62E+00	1.70E+03	3.37E+00 3.48E+00	1.59E+03
TOC*	3.30E+03 3.68E+03	6.98E+00	4.12E+03 3.85E+03	7.97E+00	2.00E+03 2.00E+03	4.00E+00	3.00E+03 3.00E+03	6.00E+00
Tc-99*	4.95E-03 5.33E-03	6.05E+00	4.90E-03 4.56E-03	5.56E+00	1.12E-02 1.16E-02	2.48E+00	1.03E-02 1.05E-02	2.26E+00
U*	2.14E+03 2.21E+03	8.70E+00	4.00E+03 3.75E+03	1.55E+01	3.01E+03 3.34E+03	9.34E+00	2.07E+03 1.82E+03	5.72E+00
pH	1.02E+01 1.02E+01	NA	9.91E+00 9.94E+00	NA	1.01E+01 1.00E+01	NA	9.72E+00 9.81E+00	NA

NA: Not Available.

* : Value less than DL or less than 5 percent above the DL.

• : Analytes with a portion of the data below 3 times the DL.

+ : Analytes with a portion of the data below 10 times the DL.

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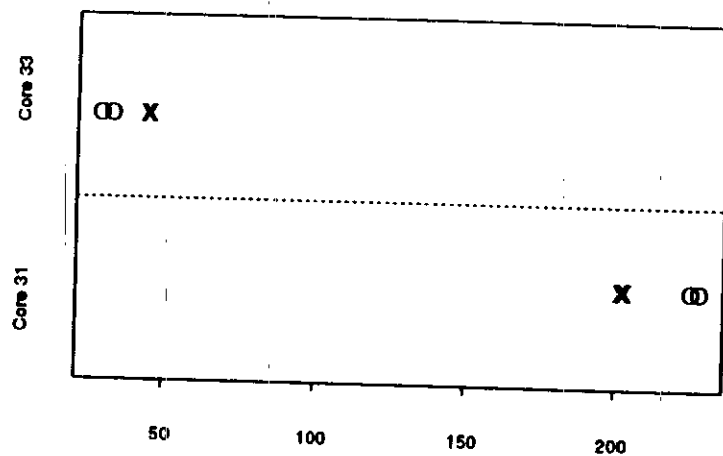
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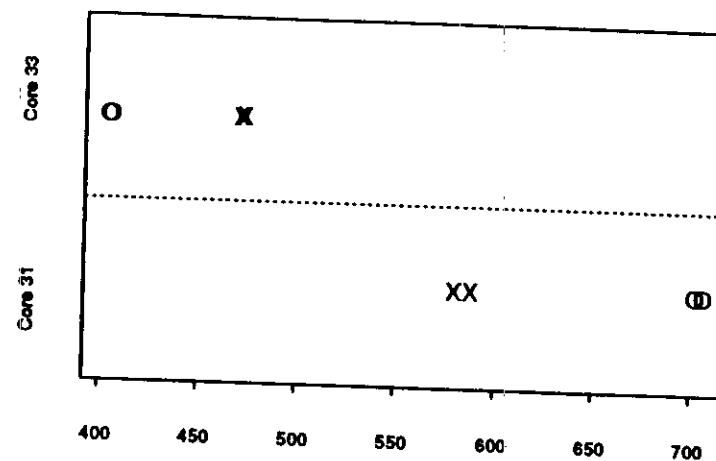
APPENDIX B
CORE COMPOSITE DATA PLOTS

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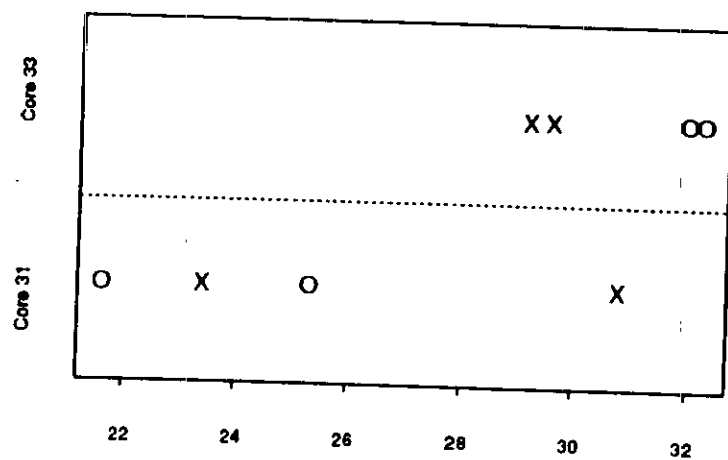
ICP.a.Ag



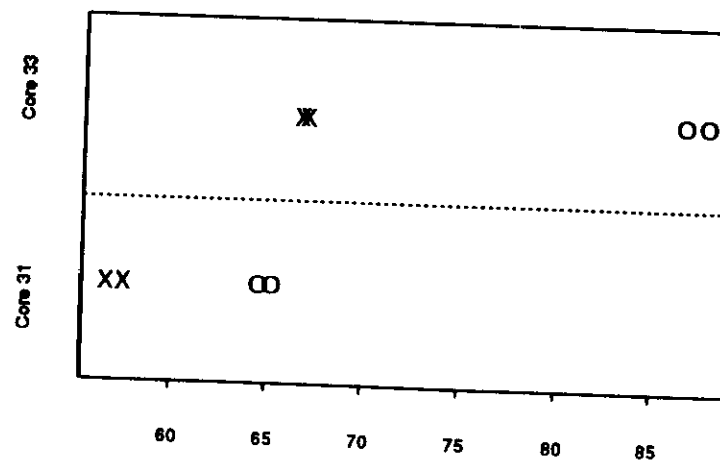
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ICP.a.B

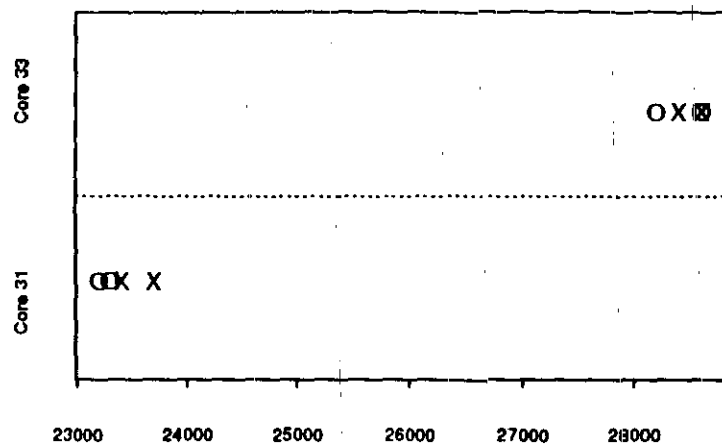


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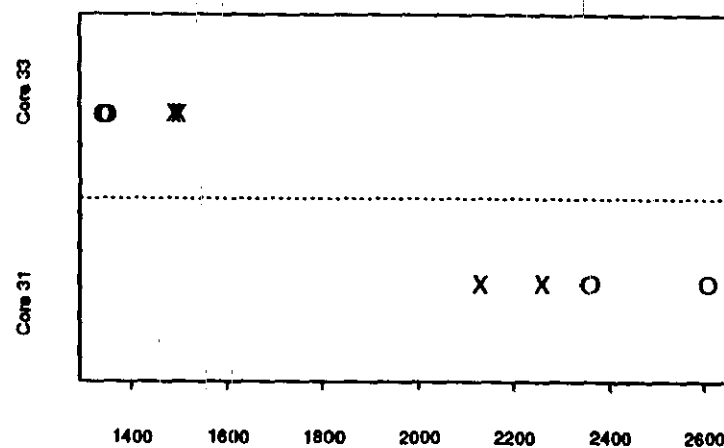


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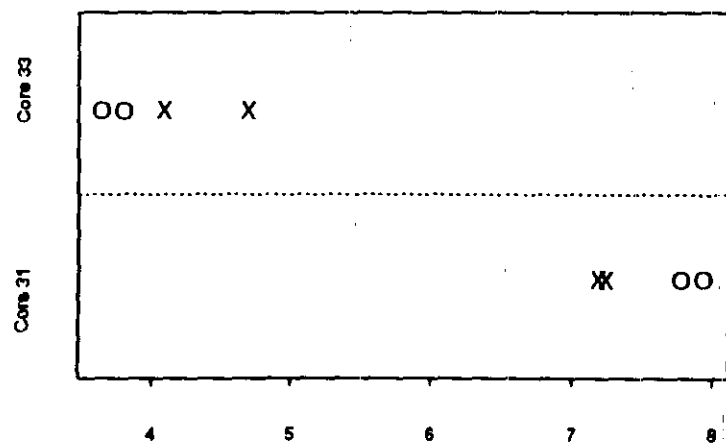
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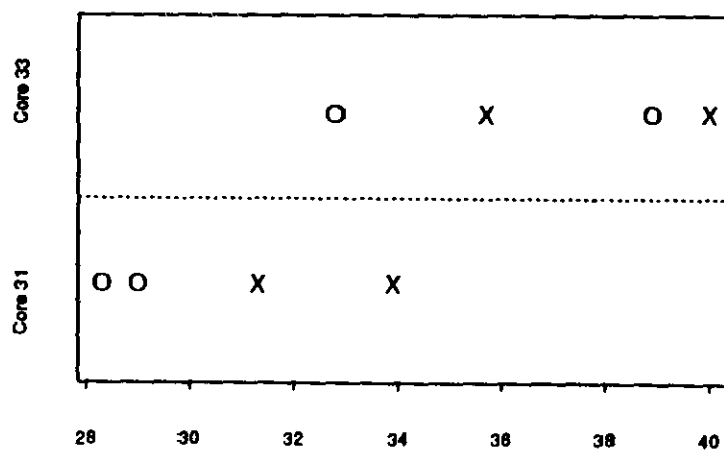
ICP.a.Ca



ICP.a.Cd

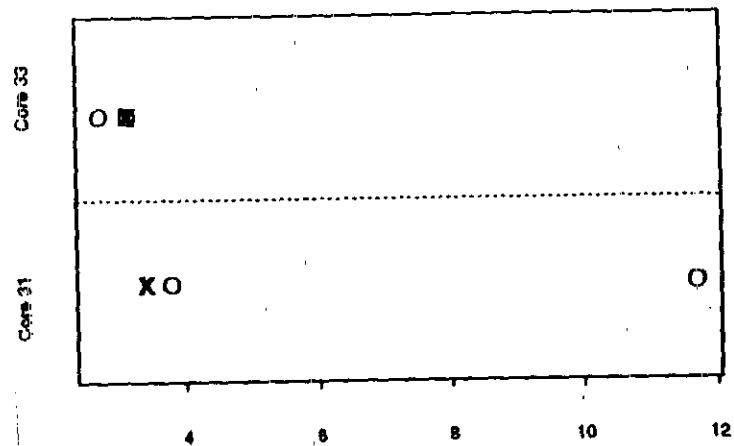


ICP.a.Ce

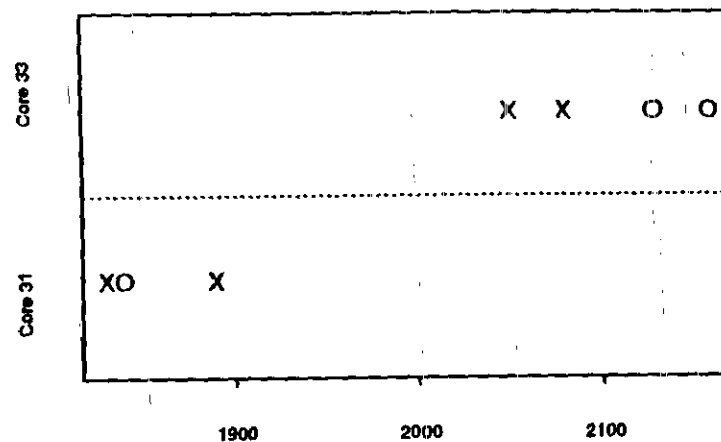


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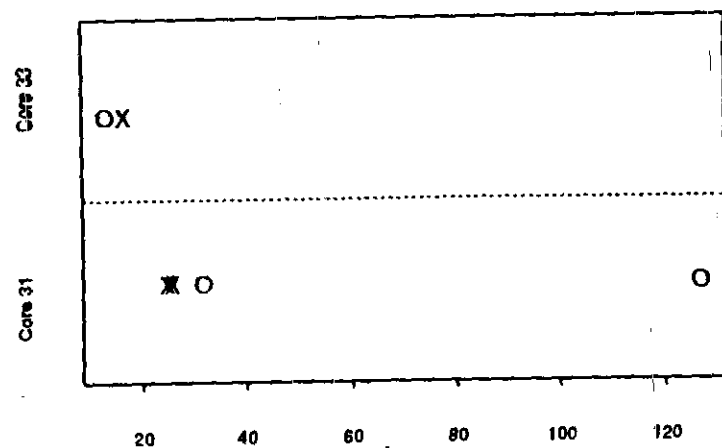
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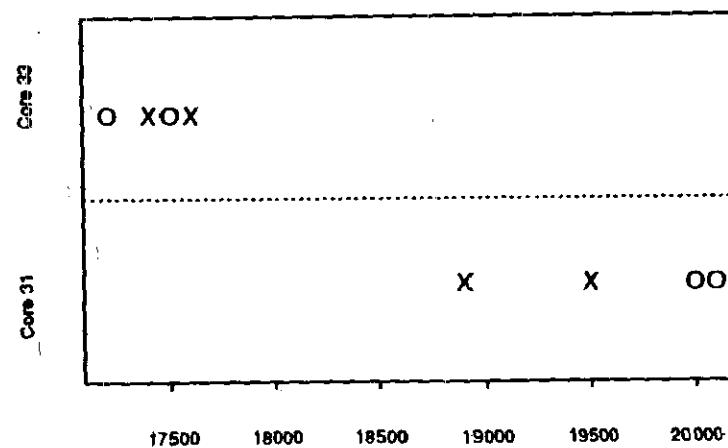
ICP.a.Cr



ICP.a.Cu



ICP.a.Fe

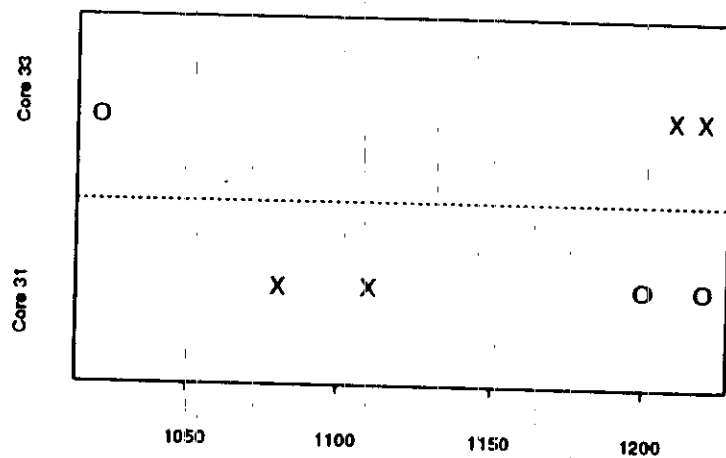


Composite 1 = O , Composite 2 = X

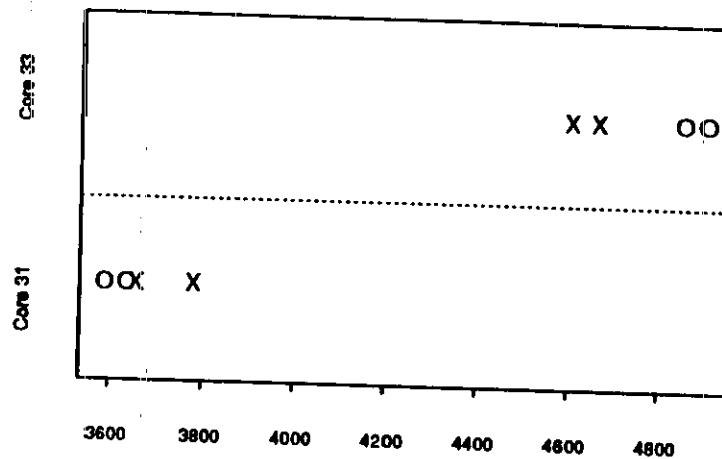
B-5

B-33

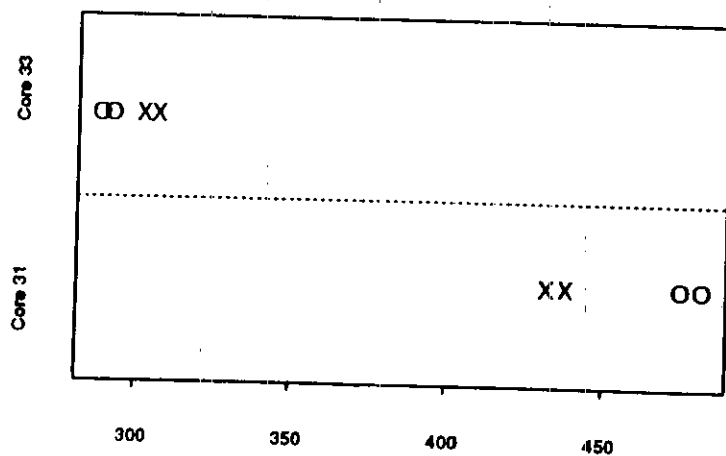
ICP.a.K



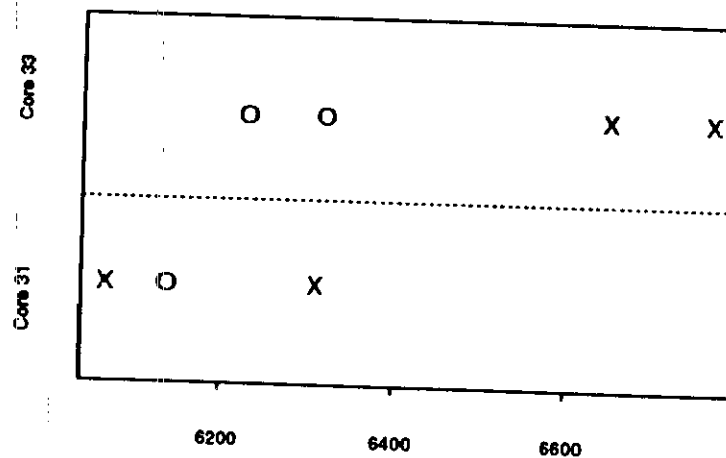
ICP.a.La



ICP.a.Mg

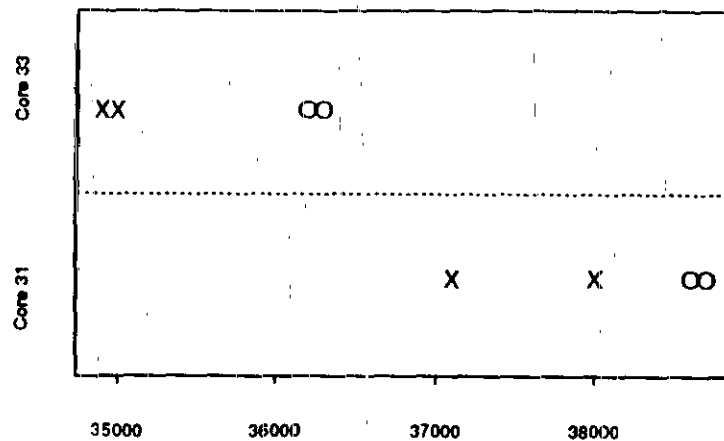


ICP.a.Mn

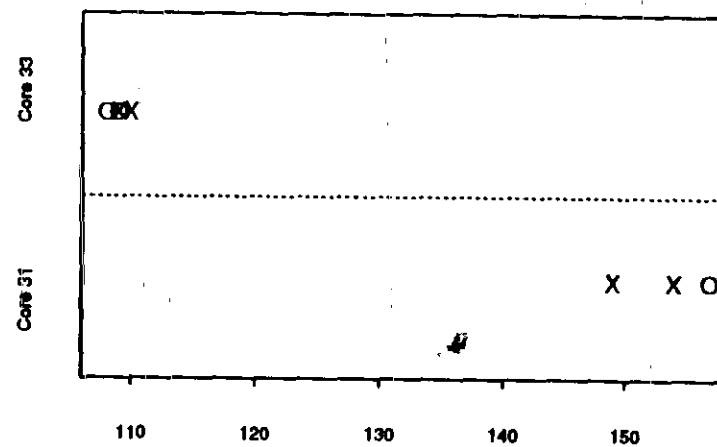


Composite 1 = O, Composite 2 = X

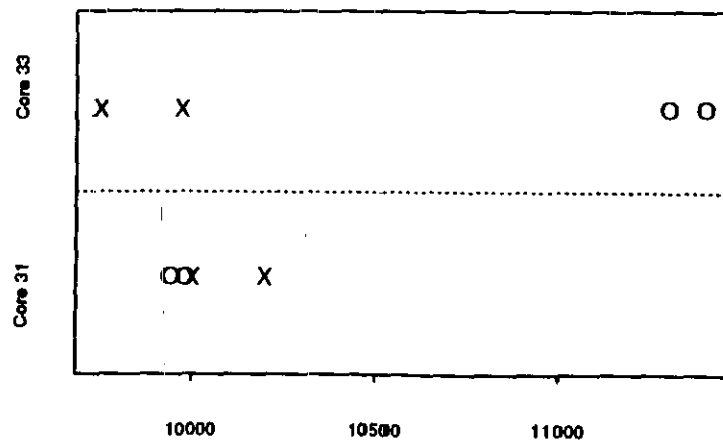
ICP.a.Na



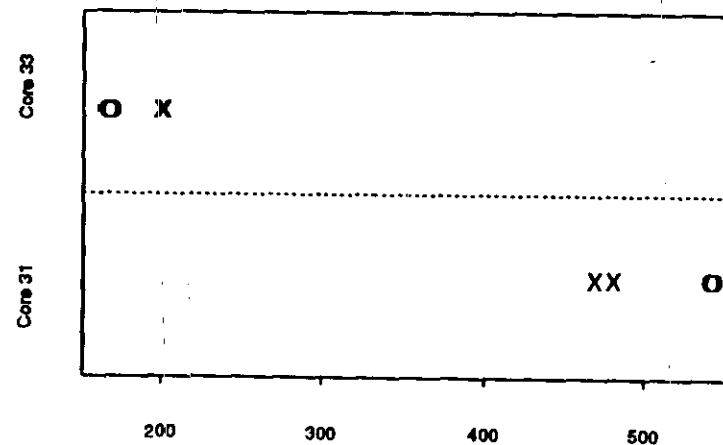
ICP.a.Ni



ICP.a.P

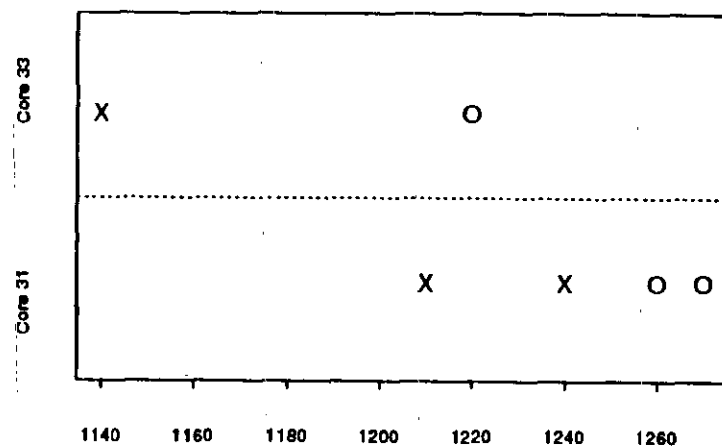


ICP.a.Pb

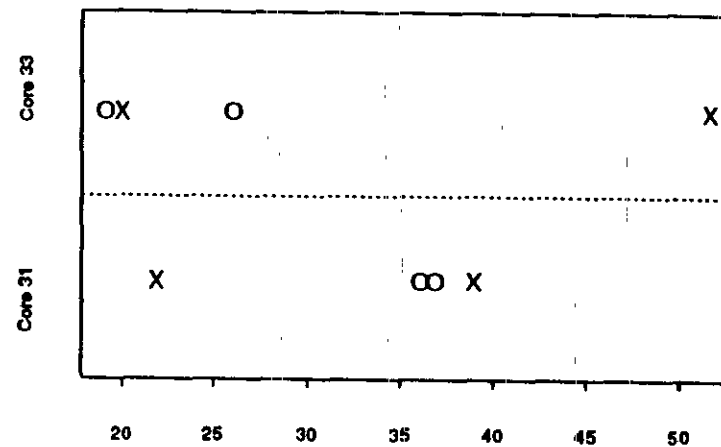


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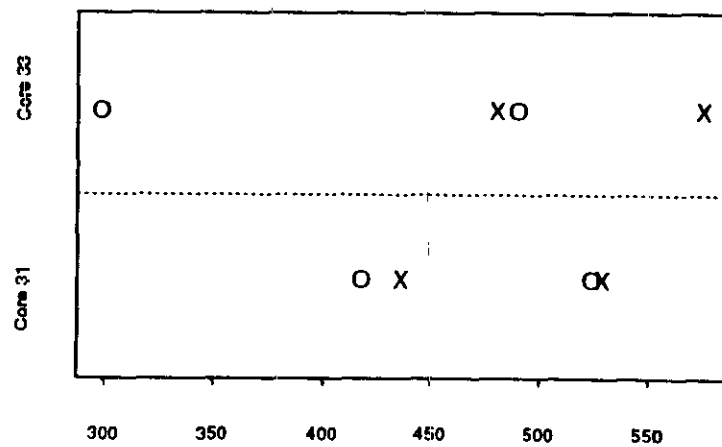
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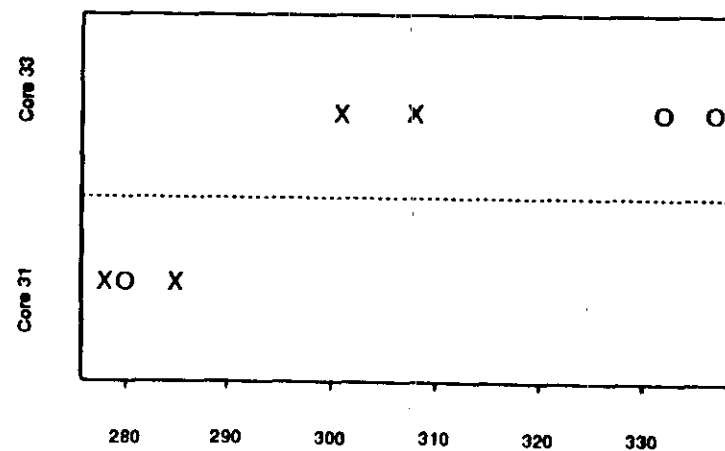
ICP.a.Sb



ICP.a.Si

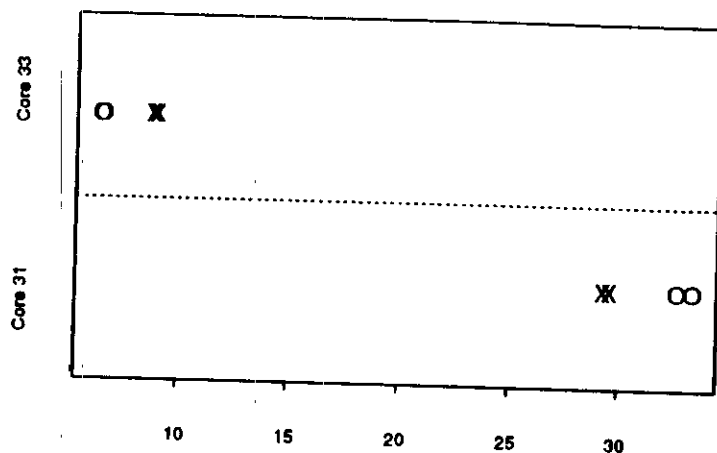


ICP.a.Sr

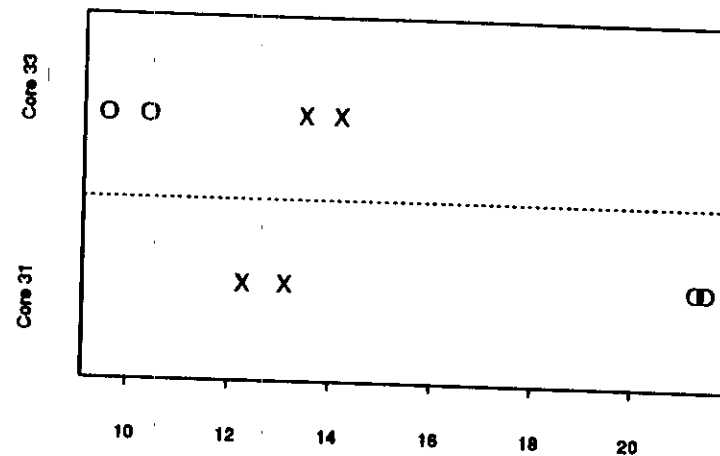


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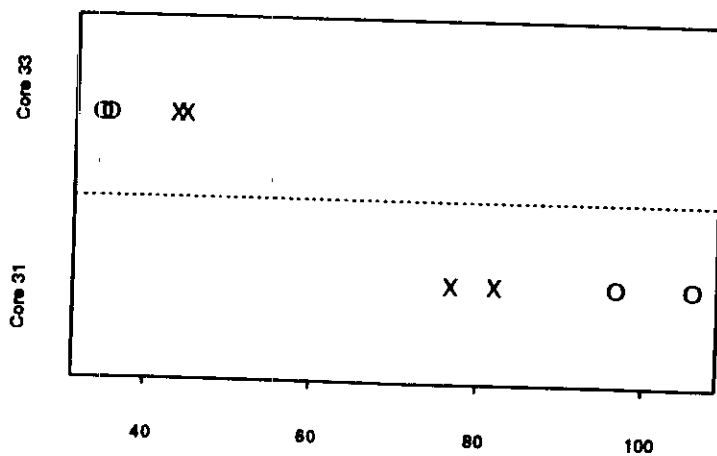
ICP.a.Ti



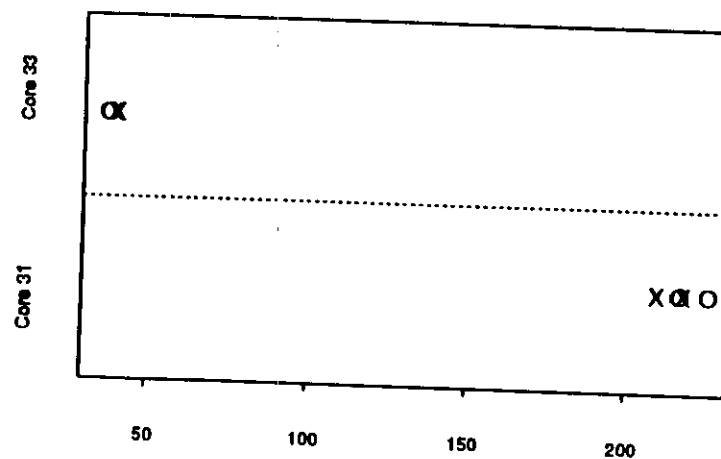
ICP.a.V



ICP.a.Zn

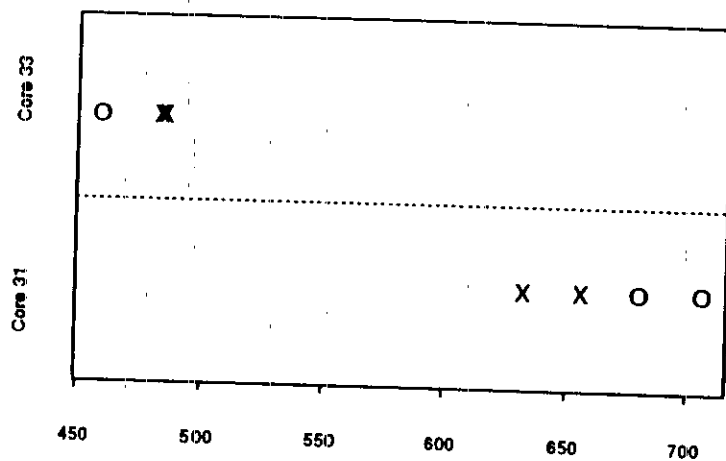


ICP.f.Ag

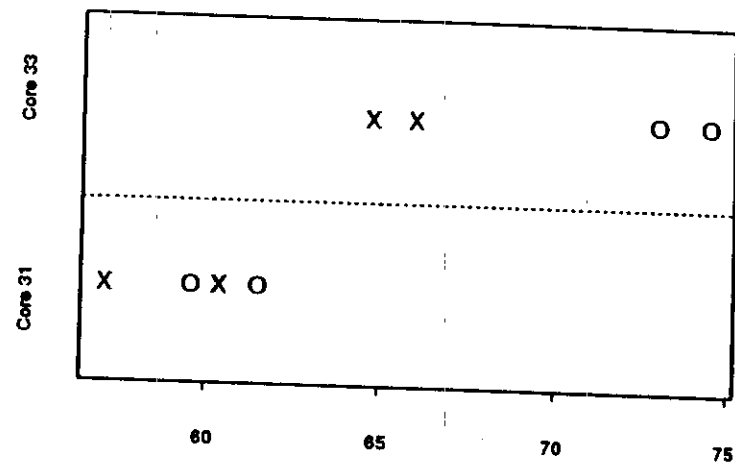


Composite 1 = O, Composite 2 = X

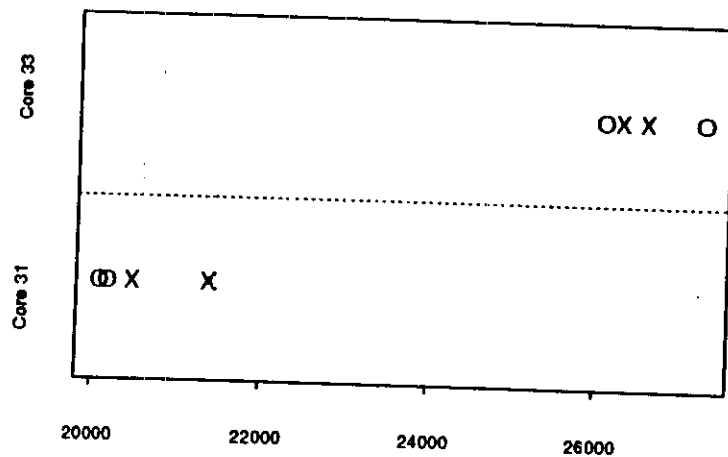
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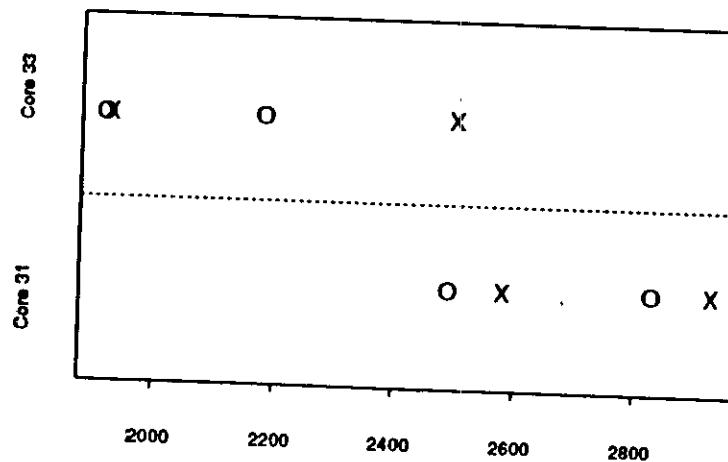
ICP.f.Ba



ICP.f.Bi



ICP.f.Ca

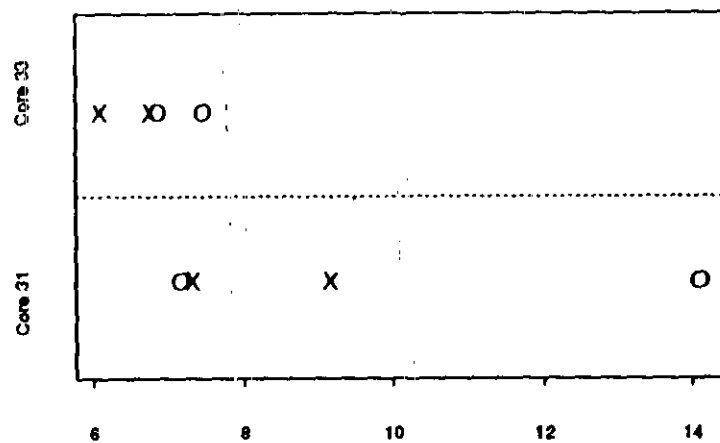


Composite 1 = O , Composite 2 = X

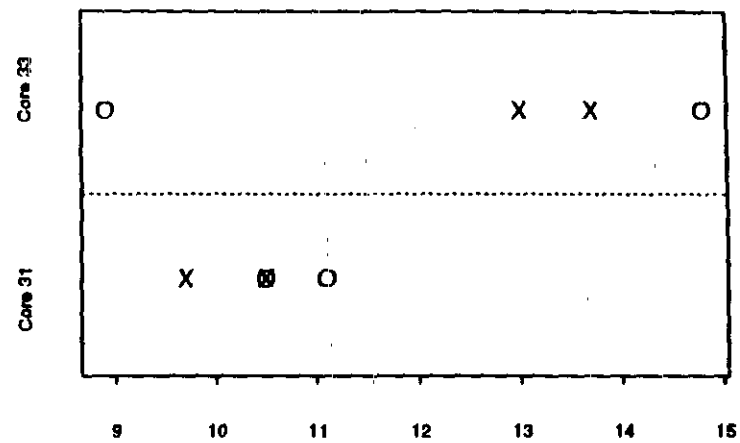
B-10

B-38

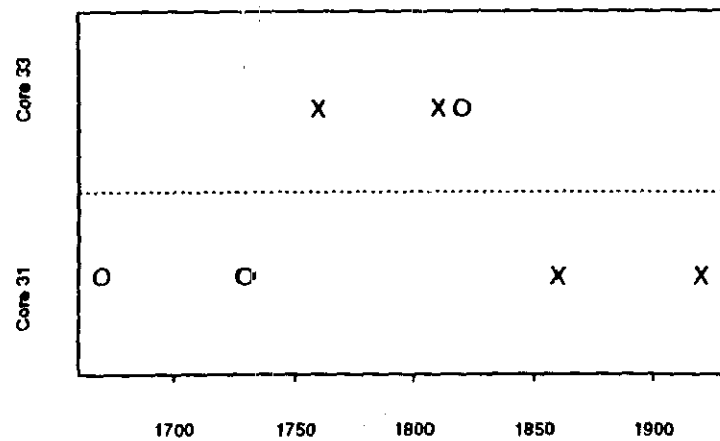
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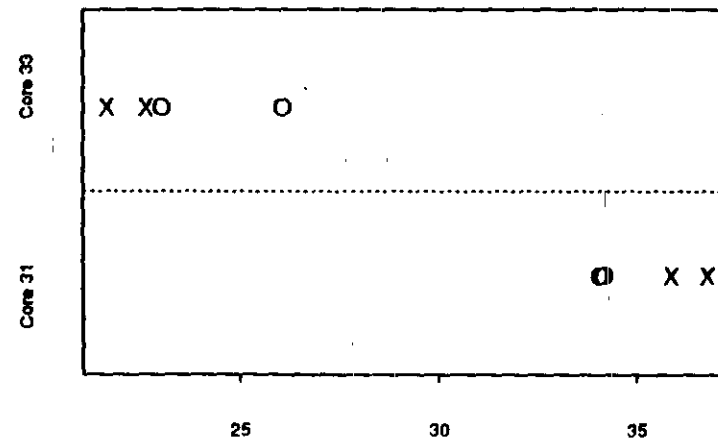
ICP.I.Co



ICP.II.Cr

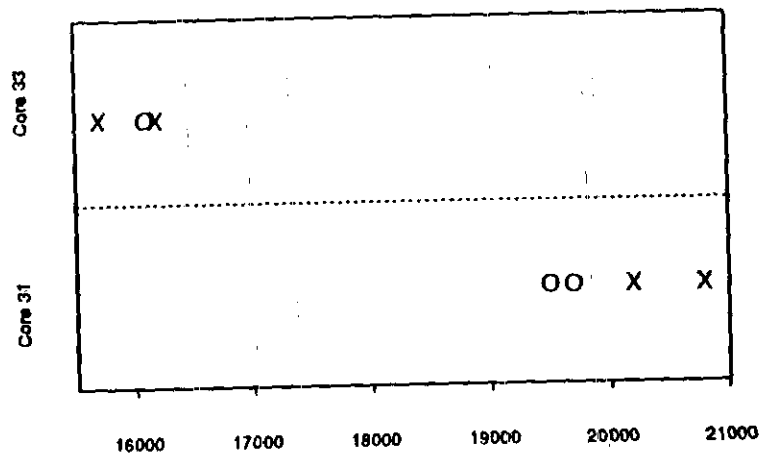


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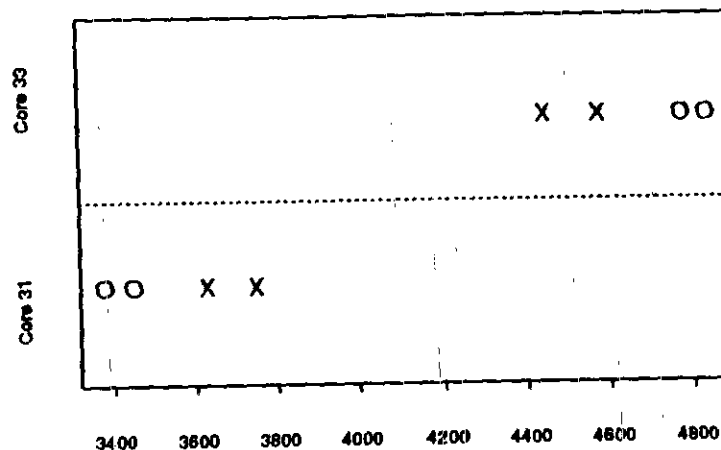


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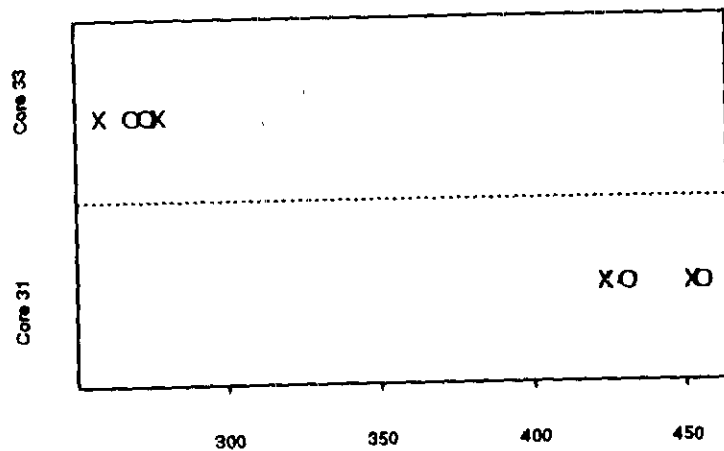
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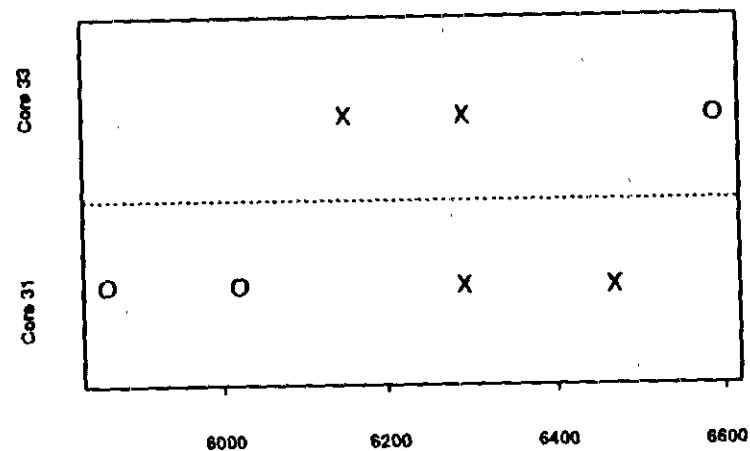
ICP.f.La



ICP.f.Mg

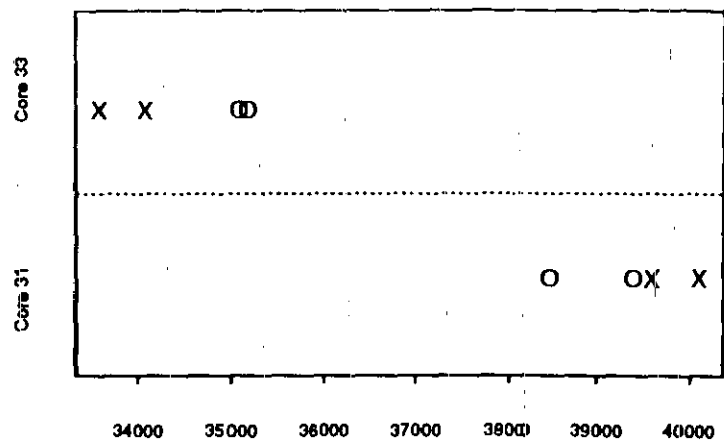


ICP.f.Mn

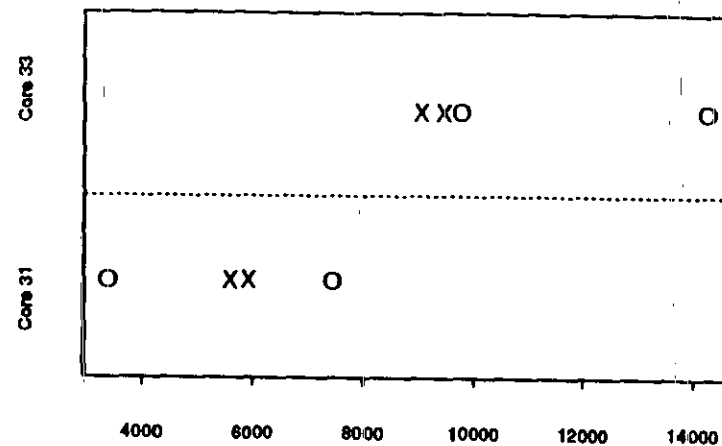


Composite 1 = O , Composite 2 = X

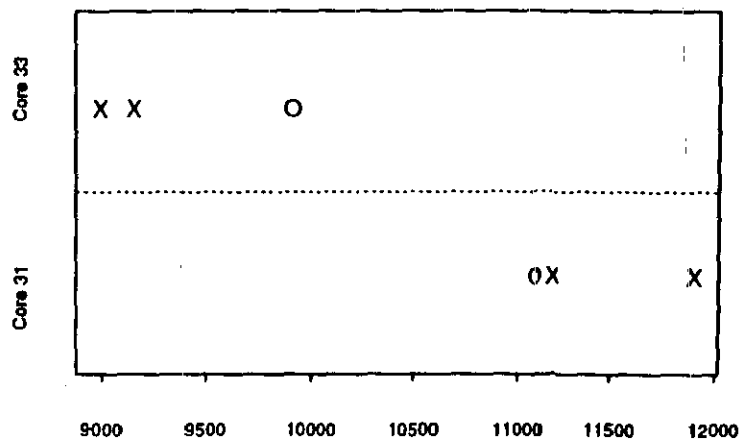
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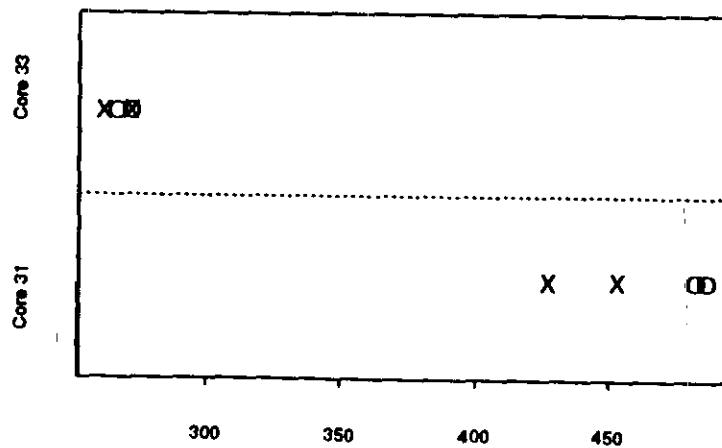
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ICP.f.P

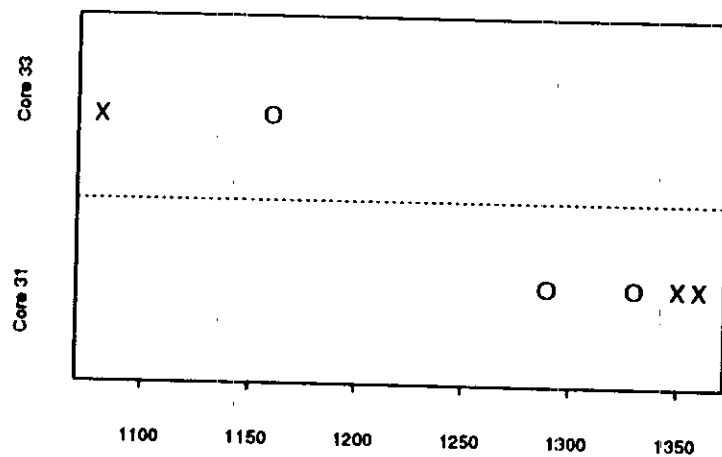


ICP.f.Pb

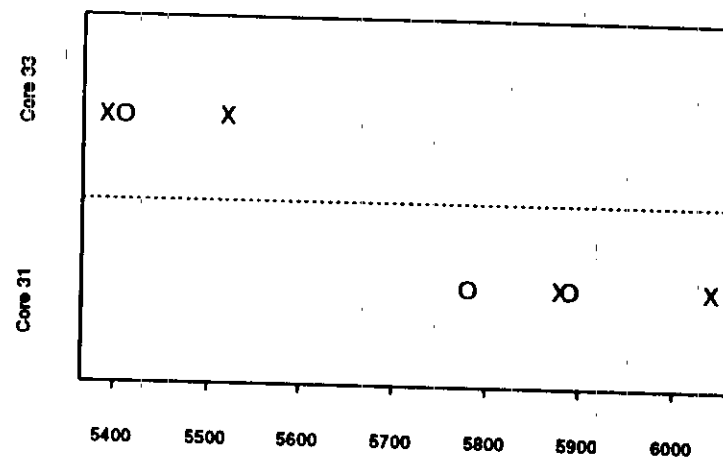


Composite 1 = O , Composite 2 = X

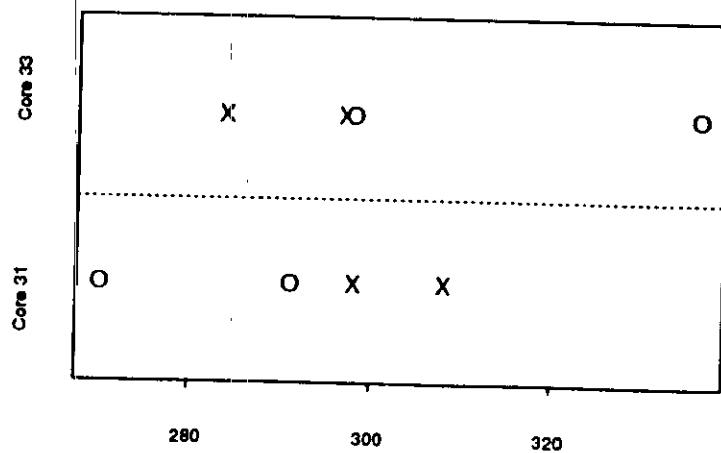
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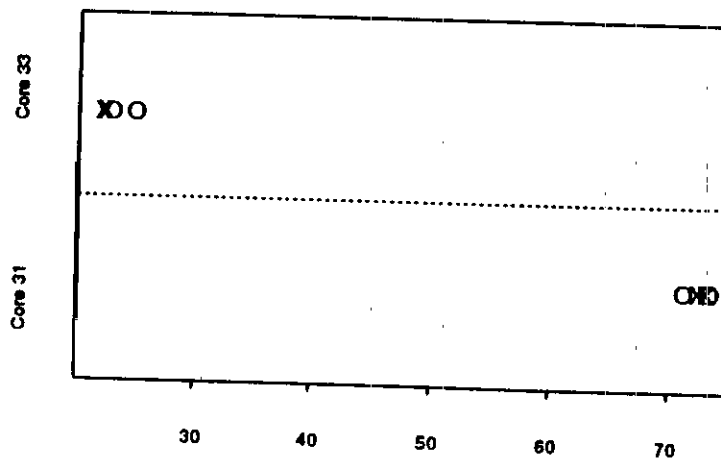
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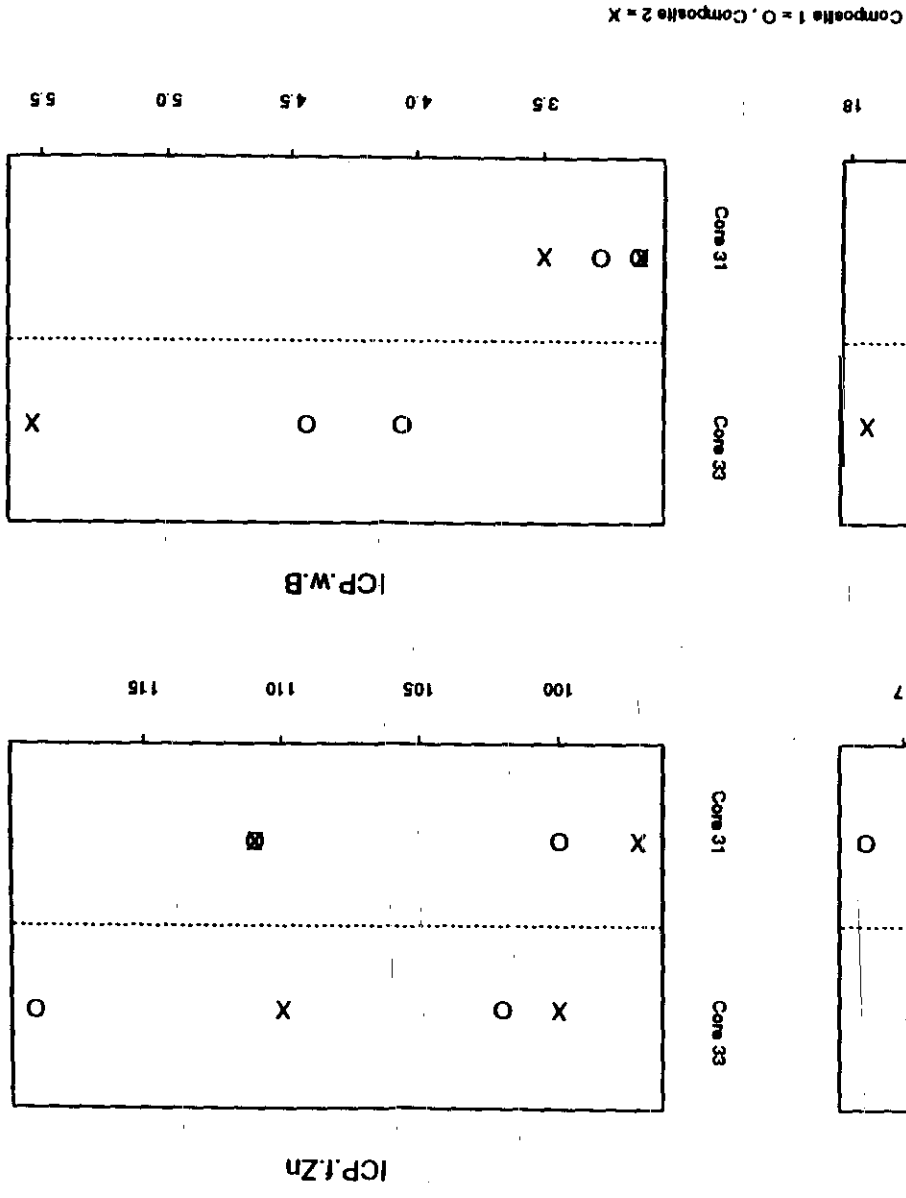
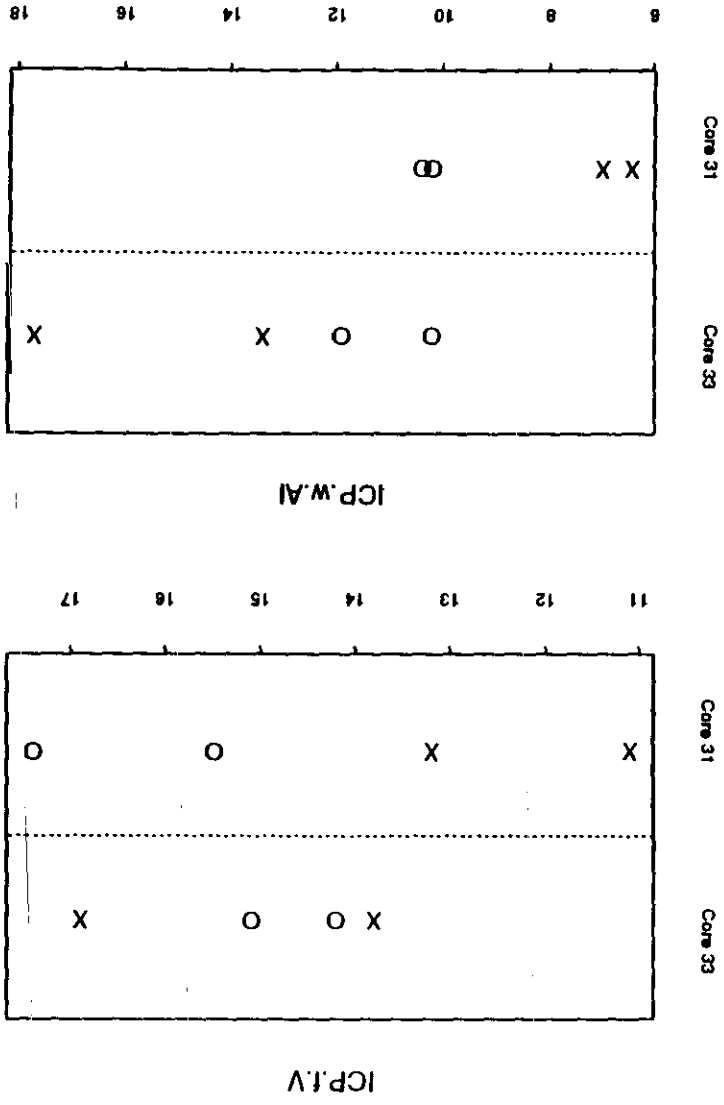
ICP.f.Sr



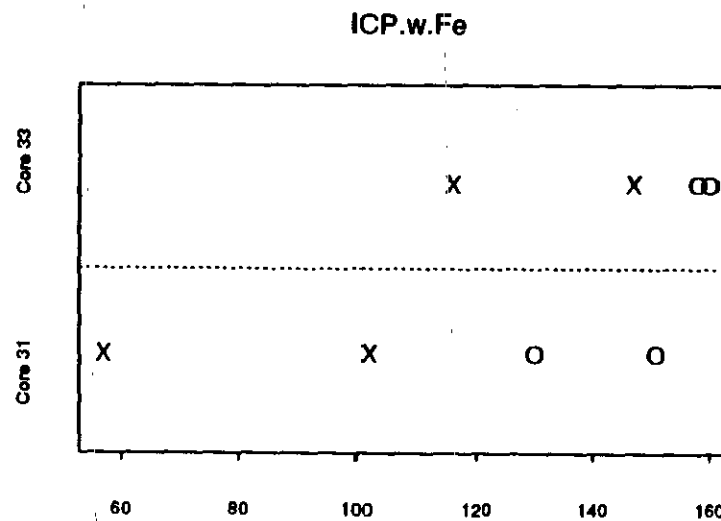
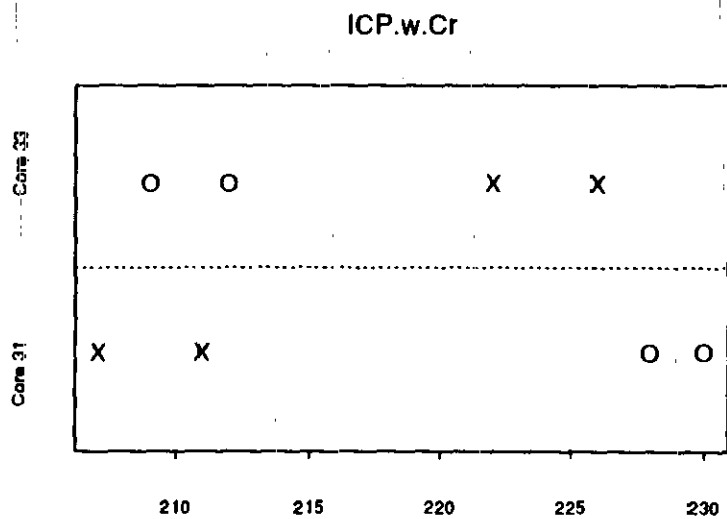
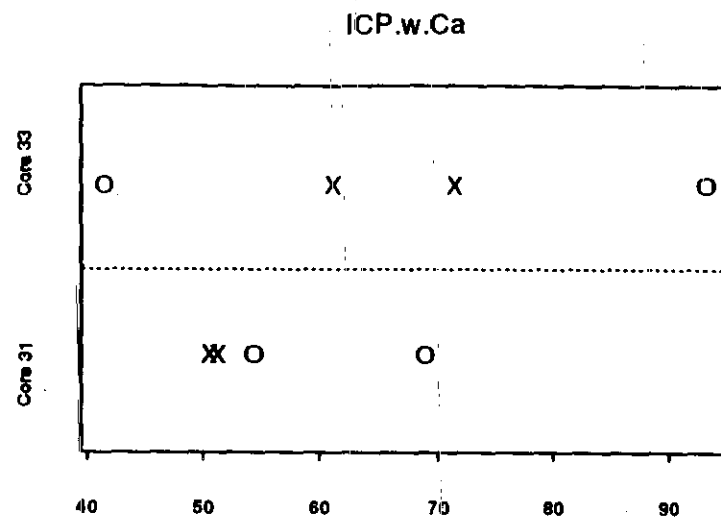
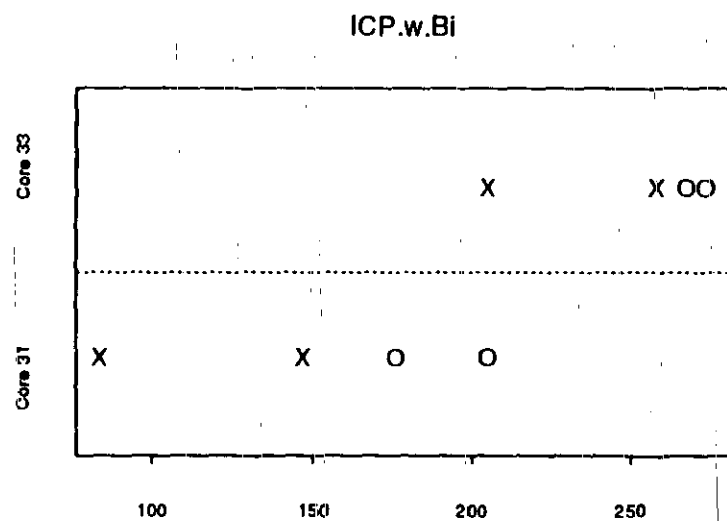
ICP.f.Ti



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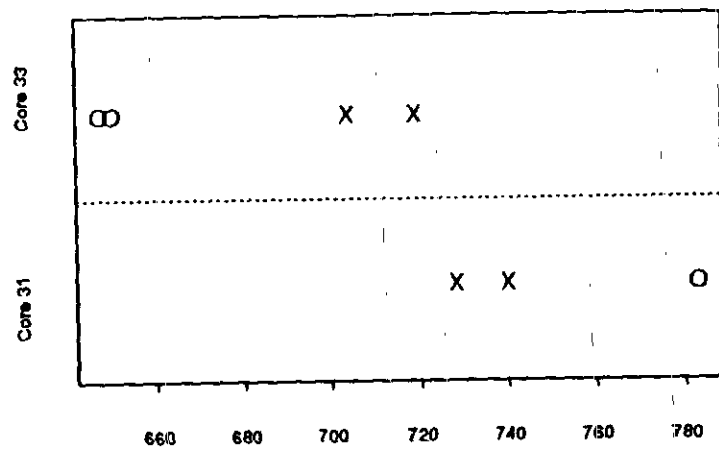


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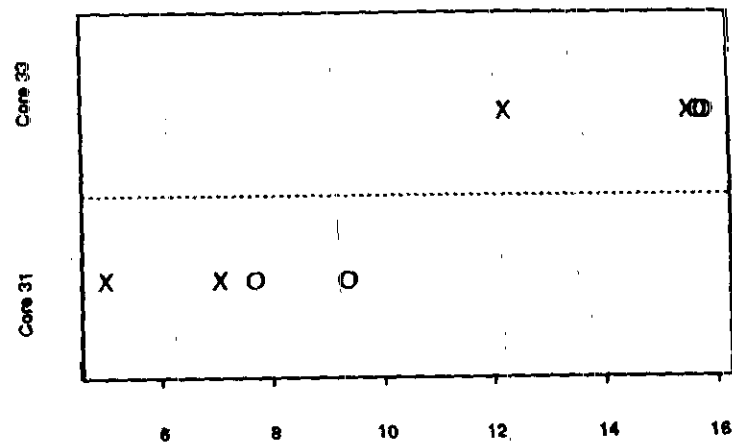


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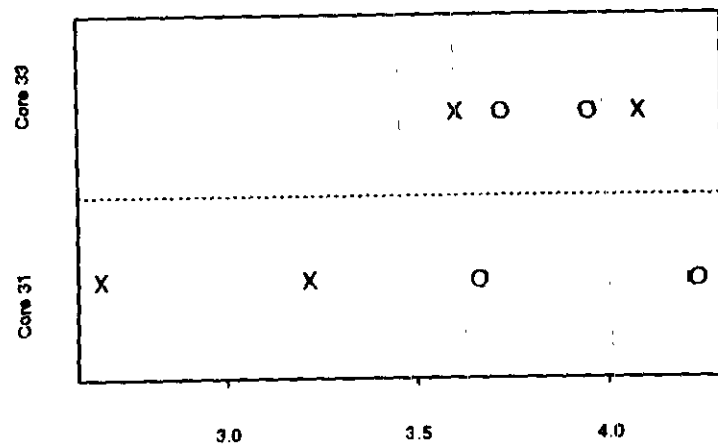
ICP.w.K



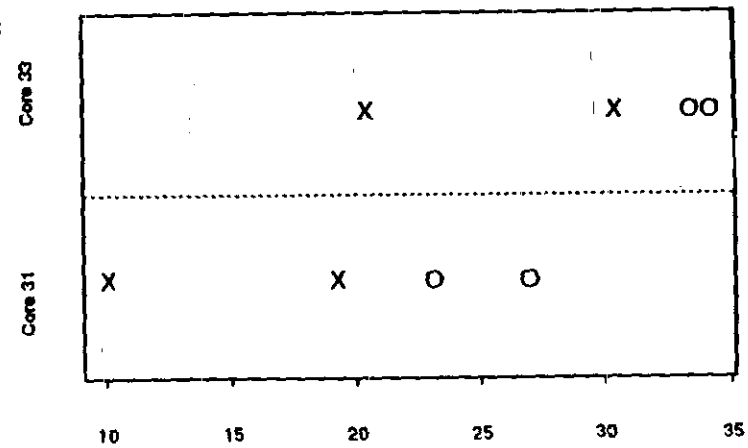
ICP.w.La



ICP.w.Mg

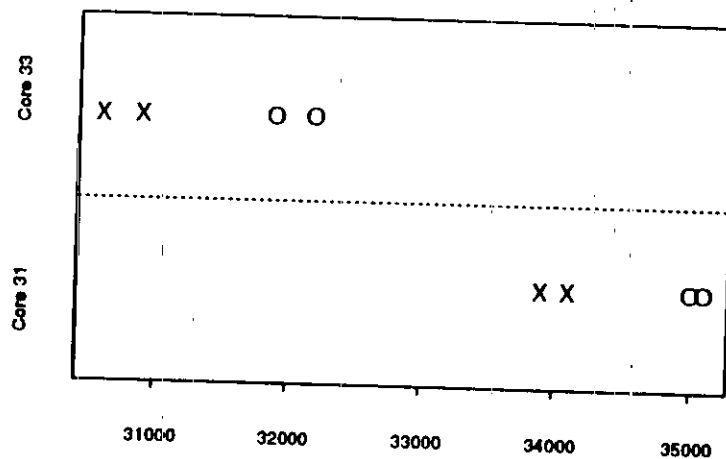


ICP.w.Mn

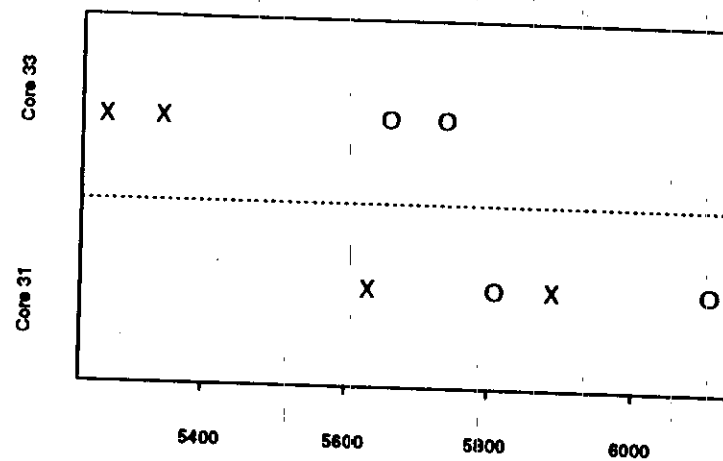


Composite 1 = O , Composite 2 = X

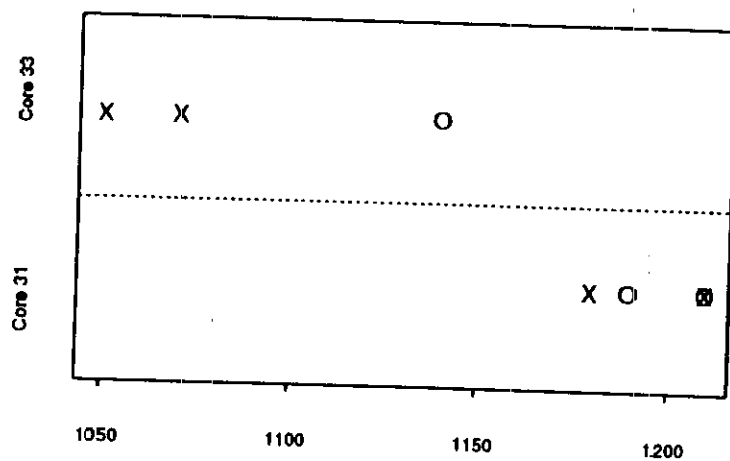
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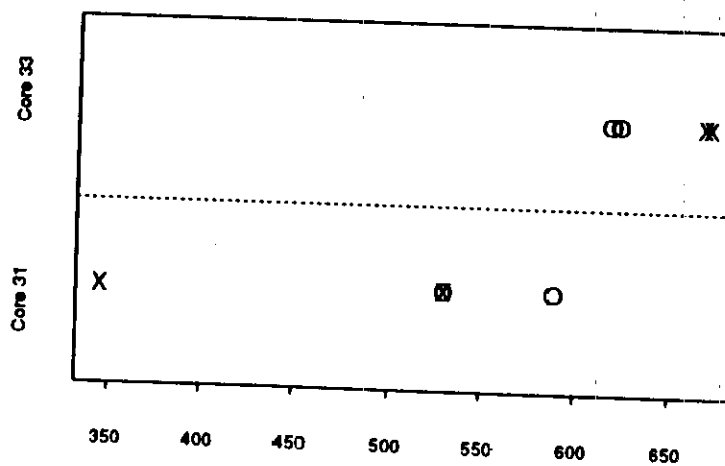
ICP.w.P



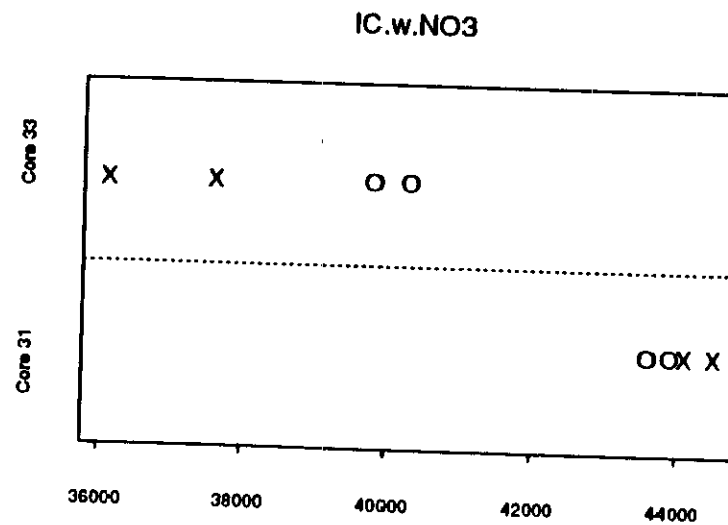
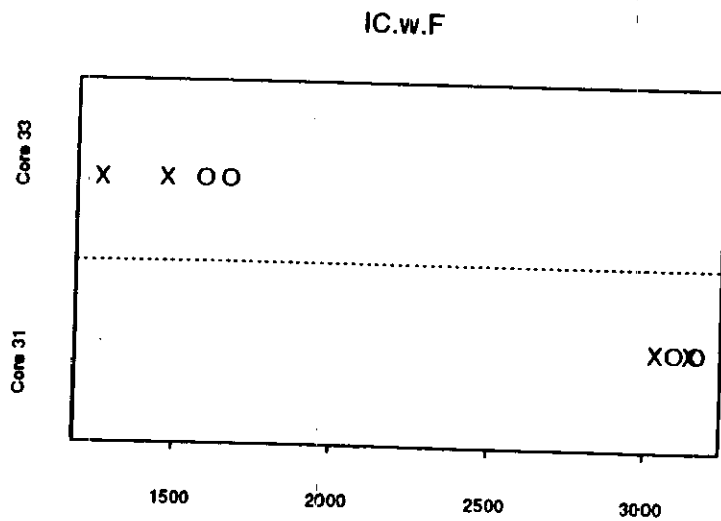
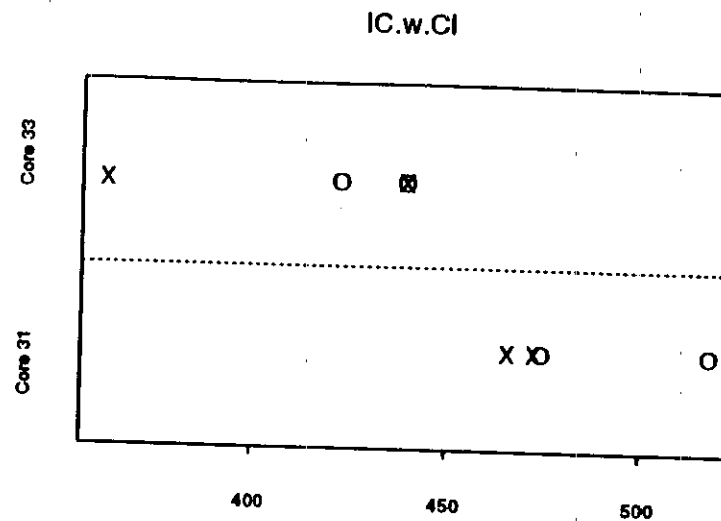
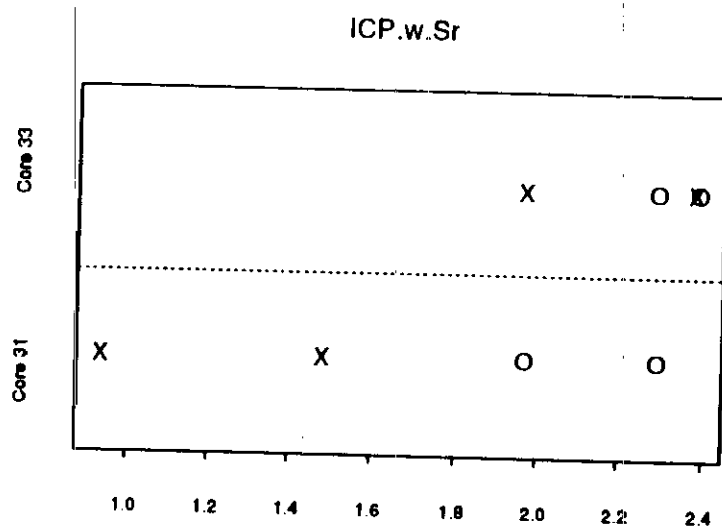
ICP.w.S



ICP.w.Si

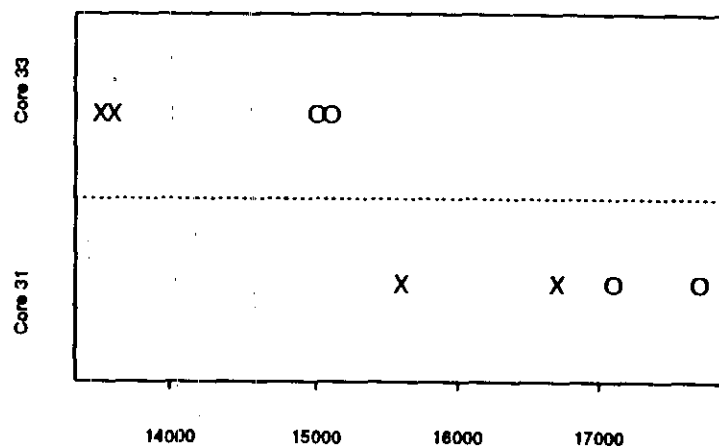


Composite 1 = O, Composite 2 = X

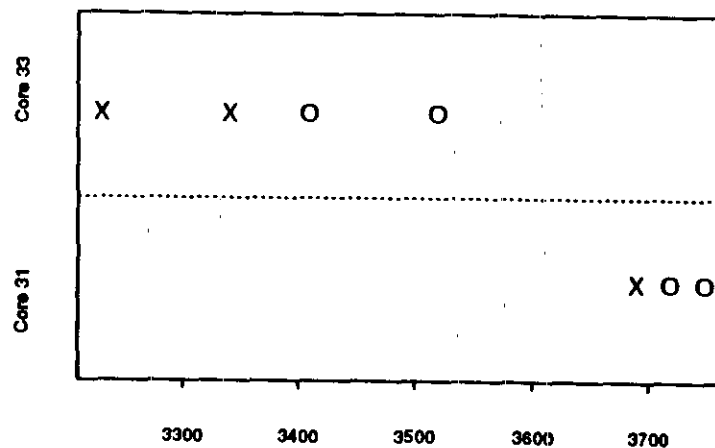


Composite 1 = O, Composite 2 = X

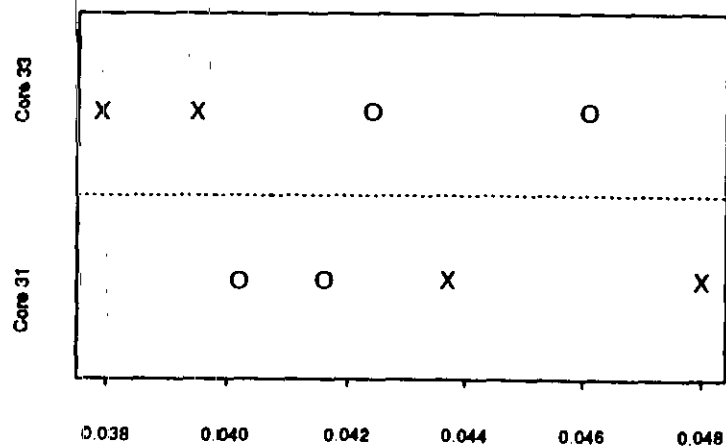
IC.w.PO4



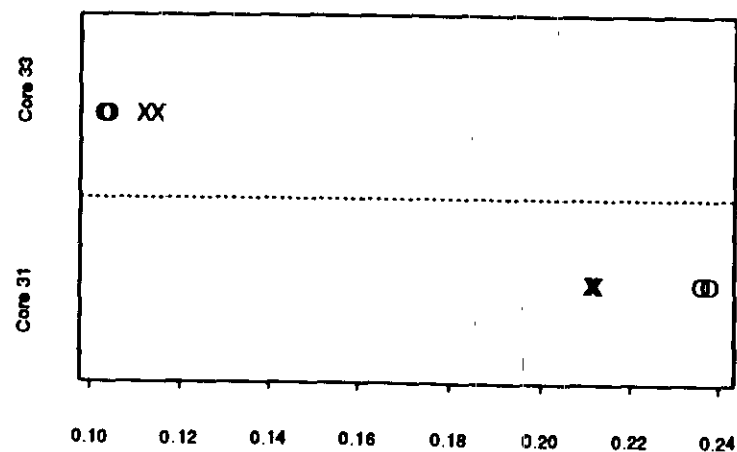
IC.w.SO4



GEA.Am-241

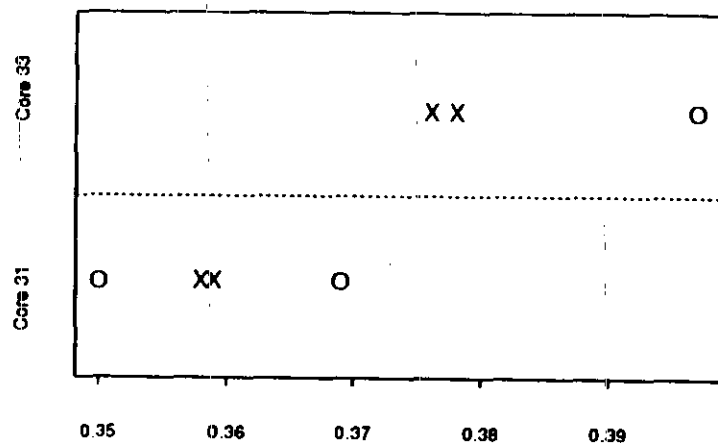


GEA.Cs-137

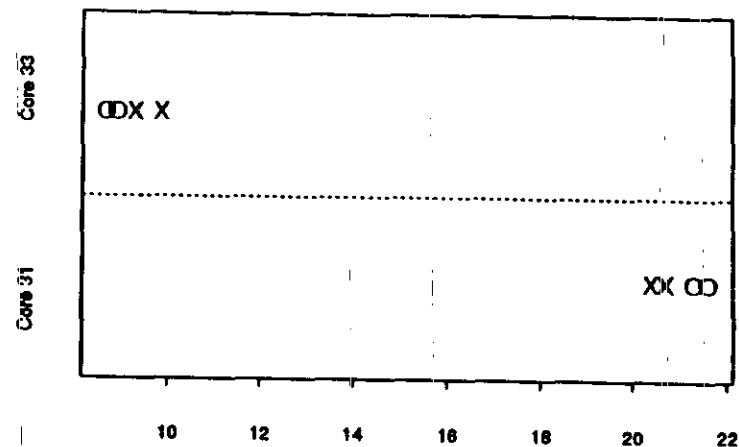


Composite 1 = O, Composite 2 = X

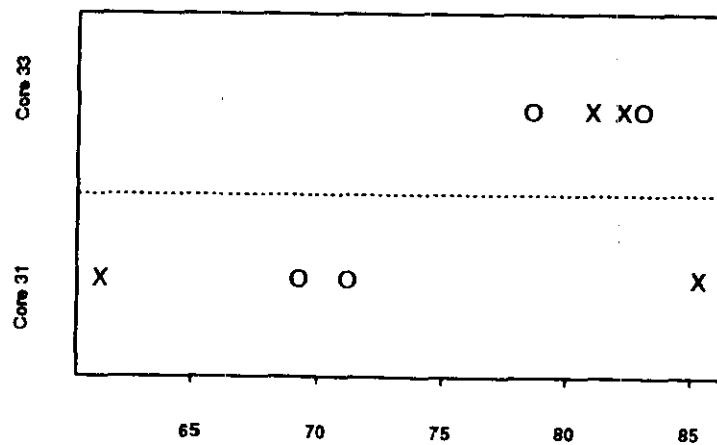
Gross.alpha



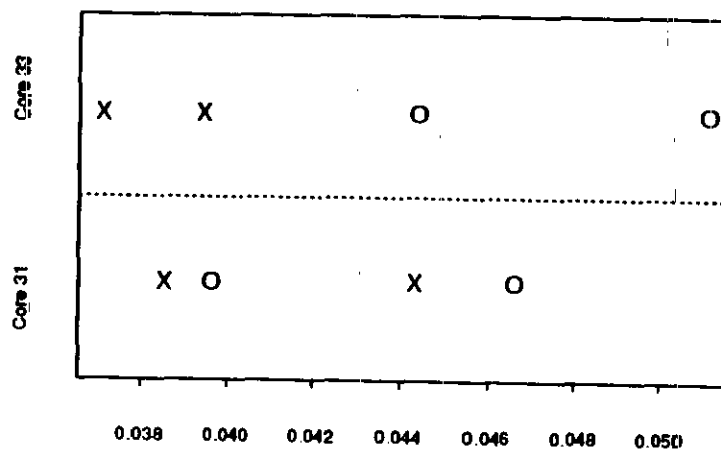
Gross.beta



TGA.Percent.H2O

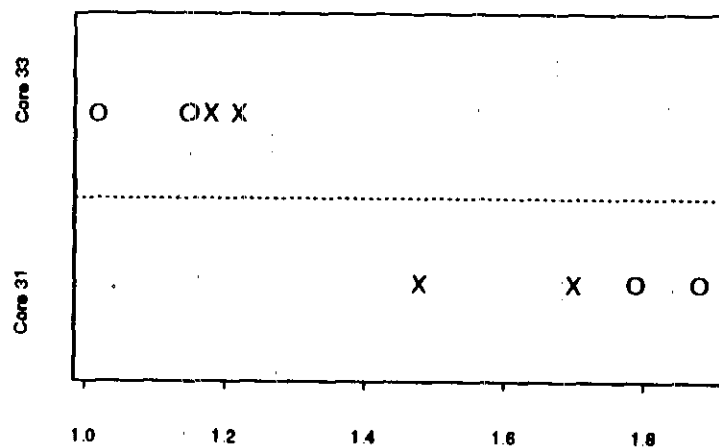


Am-241

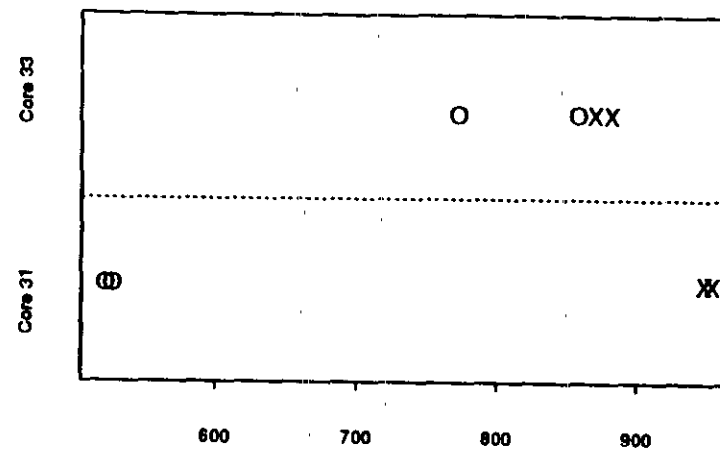


Composite 1 = O , Composite 2 = X

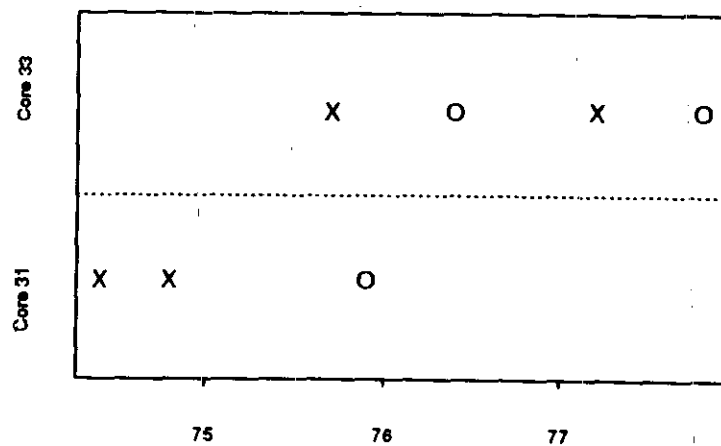
Hg



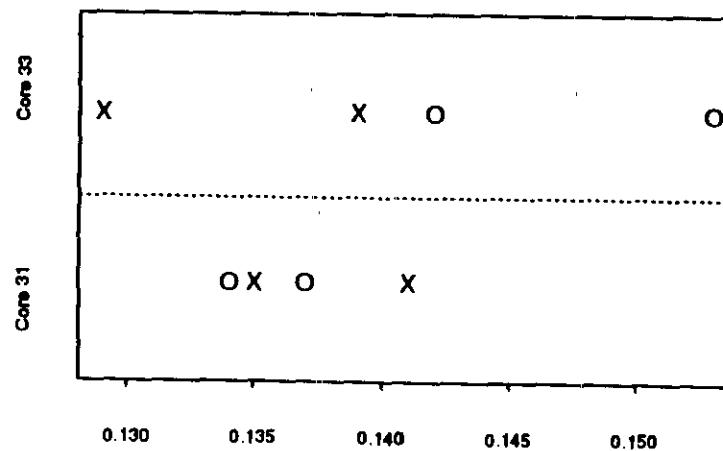
NO2



Percent.H2O

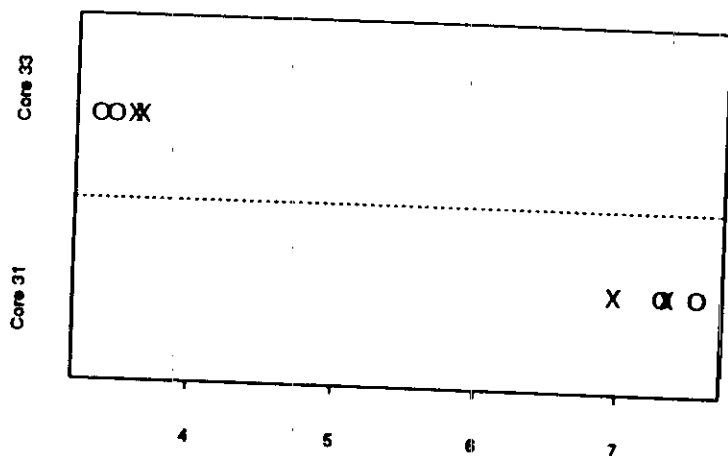


Pu-239/40

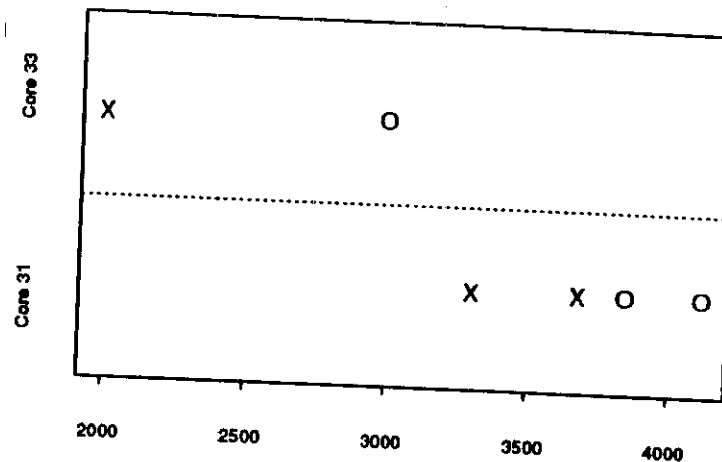


Composite 1 = O, Composite 2 = X

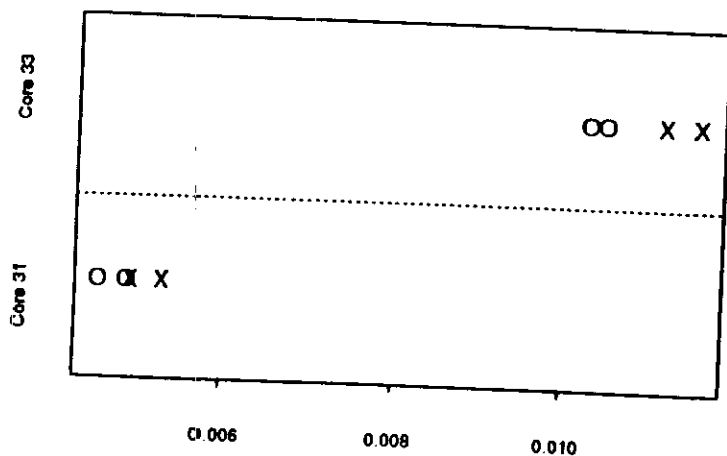
Sr-90



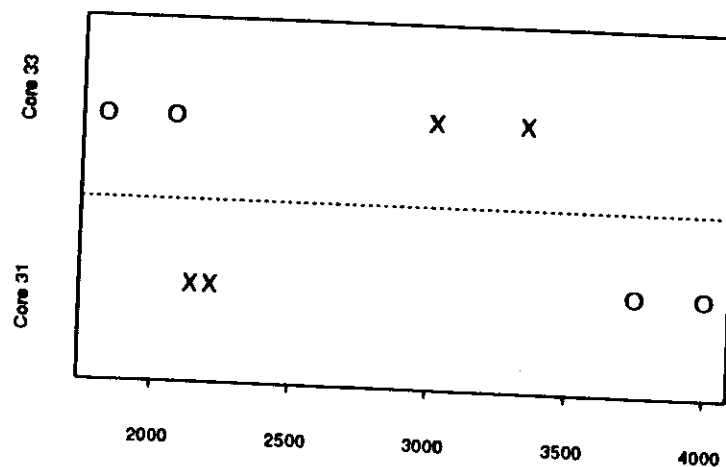
TOC



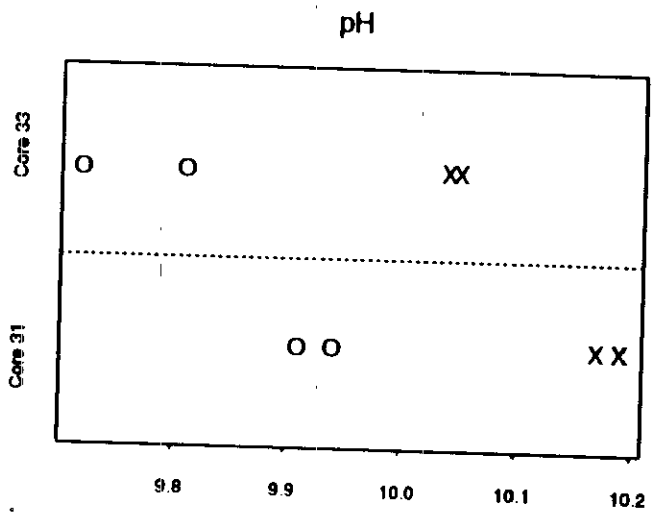
Tc-99



U



Composite 1 = O, Composite 2 = X



Composite 1 = O , Composite 2 = X

B-24

B-52

WHC-EP-0806

WHC-SD-WM-TI-650, Rev. 0

APPENDIX C

MEAN CONCENTRATION CALCULATION METHODS

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This appendix includes a description of the statistical model that describes the structure of the data from core samples taken from Tank 241-T-111. Equations are also presented for estimates of the mean concentration, the variance for the mean concentration, and the CI on the mean concentration.

The statistical model that describes the structure of the core composite data is

$$y_{ijk} = \mu + S_i + C_{ij} + A_{ijk}, \quad i=1, \dots, a, \quad j=1, \dots, b_i, \quad k=1, \dots, n_{ij}, \quad (1)$$

where

- y_{ijk} = laboratory results from the k^{th} duplicate of the j^{th} composite of the i^{th} core from the tank,
- μ = the grand mean,
- S_i = the effect of the i^{th} core (spatial effect),
- C_{ij} = the effect of the j^{th} composite sample from the i^{th} core,
- A_{ijk} = the analytical error associated with the k^{th} duplicate in the j^{th} composite from the i^{th} core,
- a = the number of cores,
- b_i = the number of composite samples in the i^{th} core and
- n_{ij} = the number of analytical results from the j^{th} composite sample in the i^{th} core.

For cores 31 and 33 there are two core composite samples (i.e., $b_i = 2$).

The variables S_i and C_{ij} are treated as random effects. It is assumed that S_i , C_{ij} and A_{ijk} are each distributed normally with mean zero and variances $\sigma^2(S)$, $\sigma^2(C)$ and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(S)$, $\sigma^2(C)$ and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML). This method applied to variance component estimation is described by Harville (1977).

WHL-SD-WH-TI-650, Rev. 0

The mean concentration of each analyte of interest in the tank was calculated using the following equation:

$$\bar{y} = \frac{1}{a} \sum_{i=1}^a \bar{y}_{i\cdot} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i\cdot}} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} (\mu + S_i + C_{ij} + A_{ijk})}{n_{i\cdot}} \quad (2)$$

where

$$\bar{y}_{i\cdot} = \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i\cdot}} \quad \text{and} \quad n_{i\cdot} = \sum_{j=1}^{b_i} n_{ij} \quad (3)$$

This mean gives the results from each core the same weight regardless of the unbalance that may exist for a particular analyte.

The variance of \bar{y} is

$$V(\bar{y}) = C_1 \sigma^2(S) + C_2 \sigma^2(C) + C_3 \sigma^2(A) \quad (4)$$

where

$$C_1 = \frac{1}{a}, \quad C_2 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i\cdot}} \right]^2 \left[\sum_{j=1}^{b_i} n_{ij}^2 \right], \quad C_3 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i\cdot}} \right] \quad (5)$$

Using $\hat{\sigma}^2(S)$, $\hat{\sigma}^2(C)$, and $\hat{\sigma}^2(A)$ (REML variance component estimates), an estimated variance of \bar{y} is

$$\hat{\sigma}^2(\bar{y}) = C_1 \hat{\sigma}^2(S) + C_2 \hat{\sigma}^2(C) + C_3 \hat{\sigma}^2(A) \quad (6)$$

The approximate degrees of freedom used for $\hat{\sigma}^2(\bar{y})$ is the number of cores with data minus one.

The lower and upper 95% CI limits (95% LL and 95% UL respectively) on the mean concentration are

$$95\% \text{ LL} = \bar{y} - t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad \text{and} \quad 95\% \text{ UL} = \bar{y} + t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad (7)$$

where $t_{.975}$ is the 0.975 quantile from a Student's t-distribution with the approximate degrees of freedom associated with $\hat{\sigma}^2(\bar{y})$.

Appendix C: Calculation and Miscellaneous Data

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ESTIMATED WASTE TANK INVENTORIES -- TOTAL 1 ST QUARTER, 1981

COMPONENT	TANK T-100		TANK T-100		TANK T-110		TANK T-111		TANK T-112		TANK T-201		TANK T-202	
	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES
Ac225	5.E-10	8.E-09	5.E-10	8.E-09	1.E-10	2.E-11	3.E-10	4.E-11	4.E-10	8.E-11	0.	0.	7.E-22	8.E-15
Ac227	8.E-11	1.E-08	5.E-10	8.E-08	2.E-10	3.E-08	4.E-10	8.E-08	1.E-09	2.E-05	0.	0.	4.E-17	8.E-13
Am241	2.E-04	2.E-01	3.E-05	2.E-02	0.E-03	7.E+00	2.E-02	2.E+01	4.E-02	3.E+01	0.	0.	8.E-05	5.E-02
Am242	1.E-14	2.E-08	1.E-15	3.E-07	4.E-14	8.E-08	1.E-11	2.E-03	2.E-12	3.E-04	0.	0.	0.	0.
Am242m	8.E-10	2.E-08	1.E-10	3.E-07	4.E-09	8.E-08	8.E-07	2.E-03	1.E-07	3.E-04	0.	0.	0.	0.
Am243	1.E-07	5.E-06	1.E-08	8.E-07	5.E-07	2.E-05	3.E-08	1.E-04	1.E-05	5.E-04	0.	0.	0.	0.
Al217	2.E-23	8.E-08	2.E-23	8.E-08	5.E-28	2.E-11	1.E-25	4.E-11	2.E-25	8.E-11	0.	0.	2.E-29	8.E-15
Ba137m	2.E-08	1.E+03	8.E-08	8.E+03	4.E-18	3.E-07	0.	0.	0.	0.	0.	0.	0.	0.
Bi210	2.E-10	4.E-12	7.E-20	2.E-12	4.E-19	1.E-11	8.E-19	1.E-11	4.E-18	8.E-11	0.	0.	8.E-22	2.E-14
Bi211	2.E-17	1.E-08	8.E-17	8.E-08	3.E-17	3.E-08	7.E-17	8.E-08	2.E-18	2.E-05	0.	0.	7.E-24	8.E-13
Bi213	2.E-18	8.E-09	1.E-18	8.E-09	4.E-21	2.E-11	1.E-20	4.E-11	1.E-20	8.E-11	0.	0.	2.E-24	8.E-15
Bi214	2.E-21	2.E-11	8.E-22	8.E-12	5.E-21	4.E-11	8.E-21	7.E-11	5.E-20	5.E-10	0.	0.	1.E-23	1.E-13
Cl4	2.E-03	1.E-01	2.E-03	1.E-01	3.E-14	2.E-12	0.	0.	8.E-38	4.E-37	0.	0.	0.	0.
Cm242	2.E-12	2.E-08	3.E-13	2.E-07	8.E-12	7.E-08	2.E-09	2.E-03	4.E-10	3.E-04	0.	0.	0.	0.
Cm244	7.E-09	1.E-04	1.E-09	2.E-05	4.E-20	8.E-18	0.	0.	7.E-38	1.E-33	0.	0.	0.	0.
Cm245	5.E-11	2.E-08	7.E-12	3.E-10	8.E-22	4.E-20	0.	0.	0.	0.	0.	0.	0.	0.
Cs135	4.E-02	7.E-03	8.E-01	1.E-01	1.E-11	2.E-12	0.	0.	8.E-37	1.E-37	0.	0.	0.	0.
Cs137	1.E-01	2.E+03	8.E-01	7.E+03	2.E-11	3.E-07	0.	0.	1.E-35	1.E-31	0.	0.	0.	0.
Fr221	2.E-10	8.E-09	2.E-19	8.E-09	4.E-22	2.E-11	1.E-21	4.E-11	1.E-21	8.E-11	0.	0.	2.E-25	8.E-15
Fr223	2.E-10	2.E-08	1.E-17	1.E-07	4.E-18	4.E-08	1.E-17	8.E-08	3.E-17	3.E-07	0.	0.	1.E-24	8.E-15
I129	2.E-02	5.E-04	1.E-01	3.E-03	4.E-12	8.E-14	0.	0.	4.E-37	8.E-39	0.	0.	0.	0.
Nb93m	7.E-08	2.E-01	1.E-08	4.E-02	8.E-08	2.E-01	8.E-08	2.E-01	1.E-05	3.E-01	0.	0.	0.	0.
Ni63	8.E-04	3.E+00	8.E-03	2.E+01	1.E-14	5.E-11	2.E-04	8.E-01	2.E-03	8.E+00	0.	0.	0.	0.
Np237	1.E-02	2.E-03	4.E-02	7.E-03	3.E-04	5.E-05	5.E-04	8.E-05	1.E-03	2.E-04	0.	0.	2.E-08	3.E-07
Np239	8.E-14	5.E-08	1.E-14	8.E-07	4.E-13	2.E-05	2.E-12	1.E-04	8.E-12	5.E-04	0.	0.	0.	0.
Pa231	3.E-07	4.E-08	1.E-08	1.E-05	8.E-07	7.E-08	2.E-08	2.E-05	5.E-08	5.E-05	0.	0.	2.E-13	2.E-12
Pa233	4.E-10	2.E-03	1.E-08	7.E-03	1.E-11	5.E-05	2.E-11	8.E-05	3.E-11	2.E-04	0.	0.	5.E-14	3.E-07
Pa234m	8.E-13	1.E-01	2.E-13	4.E-02	1.E-12	2.E-01	4.E-12	8.E-01	1.E-11	2.E+00	0.	0.	0.	0.
Pb209	8.E-18	8.E-09	8.E-18	8.E-08	2.E-20	2.E-11	5.E-20	4.E-11	8.E-20	8.E-11	0.	0.	8.E-24	8.E-15
Pb210	2.E-16	4.E-12	1.E-18	2.E-12	8.E-18	8.E-12	8.E-18	1.E-11	5.E-15	8.E-11	0.	0.	1.E-18	2.E-14
Pb211	3.E-18	1.E-08	2.E-15	8.E-08	5.E-18	3.E-08	1.E-15	8.E-08	4.E-15	2.E-05	0.	0.	1.E-22	8.E-13
Pb214	2.E-21	2.E-11	8.E-22	8.E-12	8.E-21	5.E-11	1.E-20	7.E-11	7.E-20	5.E-10	0.	0.	2.E-23	1.E-13
Pd107	1.E-02	7.E-04	5.E-02	3.E-03	2.E-12	1.E-13	0.	0.	2.E-37	1.E-38	0.	0.	0.	0.
Po210	4.E-18	4.E-12	2.E-18	2.E-12	8.E-18	8.E-12	1.E-17	1.E-11	8.E-17	8.E-11	0.	0.	2.E-20	2.E-14
Po213	2.E-27	8.E-09	2.E-27	8.E-09	8.E-30	2.E-11	2.E-29	4.E-11	2.E-29	5.E-11	0.	0.	3.E-33	8.E-15

ASSUMPTIONS USED IN THIS MODEL ARE STATED AT END OF REPORT

SD-WM-TI-057 Rev. 0-A

WMC-EP-0806

ESTIMATED WASTE TANK INVENTORIES -- TOTAL 1 ST QUARTER, 1981 -- CONTINUED														
COMPONENT	TANK T-108		TANK T-109		TANK T-110		TANK T-111		TANK T-112		TANK T-201		TANK T-202	
	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES
Po214	3 E-28	2 E-11	1 E-28	8 E-12	8 E-28	6 E-11	1 E-27	9 E-11	9 E-27	6 E-10	0.	0.	2 E-30	1 E-13
Po215	2 E-22	1 E-06	1 E-21	8 E-06	4 E-22	3 E-06	1 E-21	6 E-06	3 E-21	2 E-05	0.	0.	1 E-28	6 E-13
Po218	2 E-22	2 E-11	1 E-22	6 E-12	7 E-22	5 E-11	1 E-21	7 E-11	8 E-21	5 E-10	0.	0.	2 E-24	1 E-13
Pu238	2 E-06	8 E-03	4 E-07	2 E-03	1 E-04	4 E-01	2 E-04	7 E-01	3 E-03	1 E+01	0.	0.	5 E-07	2 E-03
Pu239	3 E-01	5 E+00	3 E-02	5 E-01	1 E+01	2 E+02	8 E+00	1 E+02	1 E+01	2 E+02	0.	0.	2 E-02	3 E-01
Pu240	6 E-03	4 E-01	7 E-04	4 E-02	3 E-01	2 E+01	4 E-01	2 E+01	7 E-01	4 E+01	0.	0.	1 E-03	6 E-02
Pu241	3 E-05	8 E-01	3 E-06	9 E-02	3 E-03	6 E+01	6 E-03	2 E+02	1 E-02	4 E+02	0.	0.	2 E-05	5 E-01
Ra223	1 E-13	1 E-06	7 E-13	8 E-06	2 E-13	3 E-06	6 E-13	6 E-06	2 E-12	2 E-05	0.	0.	5 E-20	6 E-13
Ra225	7 E-16	6 E-09	7 E-16	6 E-09	2 E-18	2 E-11	5 E-18	4 E-11	6 E-18	6 E-11	0.	0.	1 E-21	9 E-15
Ra226	7 E-14	2 E-11	3 E-14	6 E-12	2 E-13	5 E-11	3 E-13	7 E-11	2 E-12	5 E-10	0.	0.	5 E-16	1 E-13
Ru106	5 E-10	2 E-04	5 E-11	2 E-05	3 E-11	1 E-05	1 E-10	4 E-05	9 E-10	3 E-04	0.	0.	0.	0.
Sb126	3 E-09	3 E-02	3 E-10	3 E-03	4 E-09	4 E-02	4 E-09	4 E-02	5 E-09	6 E-02	0.	0.	0.	0.
Sb126m	3 E-12	3 E-02	4 E-13	3 E-03	4 E-12	4 E-02	4 E-12	4 E-02	6 E-12	6 E-02	0.	0.	0.	0.
Se79	2 E-03	9 E-03	1 E-02	5 E-02	5 E-13	3 E-12	0.	0.	5 E-38	3 E-37	0.	0.	0.	0.
Sm151	2 E-02	1 E+02	3 E-03	1 E+01	3 E-02	1 E+02	2 E-02	6 E+01	2 E-02	8 E+01	0.	0.	0.	0.
Sn126	9 E-03	3 E-02	1 E-03	3 E-03	1 E-02	4 E-02	1 E-02	4 E-02	2 E-02	6 E-02	0.	0.	0.	0.
Sr90	4 E-01	4 E+03	7 E-03	9 E+01	4 E-01	4 E+03	3 E-01	4 E+03	5 E-01	6 E+03	0.	0.	0.	0.
Tc99	2 E-01	3 E-01	1 E+00	2 E+00	6 E-11	1 E-10	0.	0.	3 E-36	5 E-36	0.	0.	0.	0.
Th227	2 E-13	1 E-06	1 E-12	8 E-06	4 E-13	3 E-06	9 E-13	6 E-06	3 E-12	2 E-05	0.	0.	8 E-20	6 E-13
Th229	1 E-10	6 E-09	1 E-10	6 E-09	3 E-13	2 E-11	9 E-13	4 E-11	1 E-12	6 E-11	0.	0.	2 E-16	9 E-15
Th230	5 E-10	2 E-09	2 E-10	8 E-10	2 E-09	1 E-08	4 E-09	2 E-08	3 E-08	1 E-07	0.	0.	6 E-12	3 E-11
Th231	5 E-11	6 E-03	1 E-11	2 E-03	8 E-11	1 E-02	2 E-10	3 E-02	7 E-10	9 E-02	0.	0.	6 E-17	8 E-09
Th234	2 E-08	1 E-01	7 E-09	4 E-02	4 E-08	2 E-01	1 E-07	6 E-01	4 E-07	2 E+00	0.	0.	0.	0.
Ti207	3 E-17	1 E-06	2 E-16	8 E-06	7 E-17	3 E-06	2 E-16	6 E-06	5 E-16	2 E-05	0.	0.	2 E-23	6 E-13
U233	2 E-06	4 E-06	2 E-06	4 E-06	3 E-09	7 E-09	8 E-09	2 E-08	1 E-08	3 E-08	0.	0.	5 E-12	1 E-11
U234	9 E-06	1 E-05	3 E-06	4 E-06	5 E-05	7 E-05	8 E-05	1 E-04	7 E-04	1 E-03	0.	0.	1 E-07	2 E-07
U235	1 E+01	6 E-03	3 E+00	2 E-03	2 E+01	1 E-02	5 E+01	3 E-02	2 E+02	9 E-02	0.	0.	2 E-05	8 E-09
U238	2 E+03	1 E-01	5 E+02	4 E-02	3 E+03	2 E-01	8 E+03	6 E-01	3 E+04	2 E+00	0.	0.	0.	0.
Y90	9 E-05	4 E+03	2 E-06	9 E+01	9 E-05	5 E+03	8 E-05	4 E+03	1 E-04	6 E+03	0.	0.	0.	0.
Zr93	9 E-01	2 E-01	1 E-01	2 E-02	1 E+00	3 E-01	1 E+00	3 E-01	1 E+00	3 E-01	0.	0.	0.	0.
Ag	2 E-08	0.	2 E-07	0.	2 E-17	0.	0.	0.	0.	0.	0.	0.	0.	0.
Al	1 E+03	0.	2 E+04	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
Ba	3 E+00	0.	2 E+00	0.	2 E-03	0.	0.	0.	6 E-03	0.	0.	0.	0.	0.
Bi	7 E+03	0.	8 E+02	0.	1 E+07	0.	1 E+07	0.	1 E+07	0.	0.	0.	3 E+02	0.
C2H303	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.

WMC-EP-0805

COMPONENT	ESTIMATED WASTE TANK INVENTORIES -- TOTAL 1 ST QUARTER, 1981 -- CONTINUED															
	TANK T-108		TANK T-109		TANK T-110		TANK T-111		TANK T-112		TANK T-201		TANK T-202			
	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES	MOLES	CURIES
C6H5O7	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
CO3	1.E+04	0.	3.E+05	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
Ca	6.E-33	0.	6.E-34	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
Ce	5.E-01	0.	2.E+01	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
Cl	4.E-06	0.	1.E-04	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
Cr	1.E+03	0.	1.E+02	0.	2.E+04	0.	3.E+04	0.	3.E+04	0.	0.	0.	2.E+02	0.	0.	0.
EDTA	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
F	3.E+04	0.	1.E+04	0.	8.E+03	0.	3.E+03	0.	3.E+03	0.	0.	0.	2.E+04	0.	0.	0.
Fe	2.E+04	0.	2.E+03	0.	4.E+05	0.	4.E+05	0.	5.E+05	0.	0.	0.	0.	0.	0.	0.
Fe(CN)6	3.E+01	0.	3.E+00	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
HEDTA	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
K	3.E+02	0.	3.E+01	0.	0.	0.	0.	0.	0.	0.	0.	0.	2.E+04	0.	0.	0.
La	0.	0.	0.	0.	0.	0.	8.E+03	0.	8.E+02	0.	0.	0.	1.E+02	0.	0.	0.
Mn	0.	0.	0.	0.	0.	0.	2.E+04	0.	2.E+03	0.	0.	0.	3.E+02	0.	0.	0.
NO2	3.E+02	0.	2.E+04	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
NO3	2.E+04	0.	3.E+05	0.	3.E+04	0.	0.	0.	4.E-28	0.	0.	0.	1.E+05	0.	0.	0.
Na	4.E+05	0.	1.E+06	0.	8.E+04	0.	0.	0.	5.E-07	0.	1.E+01	0.	2.E+05	0.	0.	0.
Ni	6.E-03	0.	6.E-04	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
OH	6.E+04	0.	8.E+03	0.	1.E+06	0.	2.E+06	0.	2.E+06	0.	1.E+01	0.	5.E+04	0.	0.	0.
PO4	1.E+05	0.	5.E+04	0.	1.E+07	0.	1.E+07	0.	1.E+07	0.	0.	0.	3.E+03	0.	0.	0.
Pb	4.E-11	0.	3.E-10	0.	6.E-11	0.	1.E-10	0.	4.E-10	0.	0.	0.	9.E-18	0.	0.	0.
SiO3	7.E+01	0.	4.E+03	0.	2.E-05	0.	0.	0.	6.E-30	0.	0.	0.	0.	0.	0.	0.
SO4	3.E+03	0.	8.E+04	0.	2.E-03	0.	0.	0.	6.E-03	0.	0.	0.	3.E+02	0.	0.	0.
Sr	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.
ZrO	2.E+03	0.	3.E+02	0.	4.E-01	0.	3.E-01	0.	4.E-01	0.	0.	0.	0.	0.	0.	0.

C-3

WMC-EP-0806

WHC ENVIRONMENTAL INDUSTRIAL HYGIENE FIELD MONITORING

Page 1 of 2

1. Work Package No.: ZW-94-00244-W

2. Contact: BUTCH HALL

3. Building/Facility: 272-WA TANK FARM

4. Area: 2-W

5. Monitoring Date: 3-5-94

6. Room/Location/Site No. T-FARM

7. Monitoring By: Ean D. Church

8. PPE (Resp. Prot. & Prot. Clothing)

RWP-CS-009

9. Engineering Controls/Work Practices:

ALARA

10. Instrument Type

11. Instrument ID Nos.

12. Calibration Data. (Date, Time)

MX251

9308082-094

3-5-94 0700

OVM580B

37025-255

3-5-94 0700

13. Monitoring Type

14. Time

15. Location

16. Activity

17. Results

O₂

0940

111-T

SUPERNATANT SAMPLES 20.9%

LEL

1

111-T

SUPERNATANT SAMPLES 0.0%

ORGANICS

1040

111-T

SUPERNATANT SAMPLES *9.2 PPM

ORGANICS

0900 - 10:40

111-T

BREATHING ZONE 0.0 PPM

N/A

[illegible]

**Westinghouse
Hanford Company**

WHC-EP-0806

3-3

**Internal
Memo**

From: West Tank Farms Operations
Phone: 372-3919 T4-08
Date: March 3, 1994
Subject: TANK T-111

94-003


To: D. A. Turner R2-78

cc: H. Babad R2-78 J. W. Lentsch -78
D. C. Board S1-57 D. M. Lucoff R2-36
V. C. Boyles R1-49 G. J. Miskho R2-2
D. A. Bragg R1-49 D. J. Newland R2-5
R. G. Brown R2-11 R. Ni S5-07
R. J. Cash R2-78 J. W. Osborne R2-78
G. M. Christensen H4-21 M. A. Payne R2-31
C. DeFigh-Price R2-31 M. Plys H2-62
A. J. Duckett H0-39 A. Postma H4-61
G. T. Dukelow R2-78 T. E. Rainey R4-02
J. C. Fulton R2-31 R. E. Rayond R2-74
J. M. Grigsby H2-62 J. P. Sederburg R2-
G. A. Hanson R2-08 M. H. Shannon H4-61
J. O. Honeyman R2-52 M. J. Sutey R1-49
M. N. Islam R2-08 O. S. Wang R2-78
G. D. Johnson R2-178 N. W. Kirch R2-11
R. D. Wojtasek R2-36 J. L. Lee R2-36
JHW File/LB

References: (1) Internal Memo, Organic Tank Safety to J. H. Wicks, et al., "Tank T-111" dated February 28, 1994

In accordance with your recommendation in reference 1, Tank T-111 has been placed on the organics tanks Watch List. Operational Specifications for Watch List Tanks (OSD-T-151-00030) has been changed to add tank T-111 to the Organic Salt List in Appendix A. Notification of this change was included in the Daily Operating Report (DOR) on 3/3/94.

If you have any questions, please call David P. Reber (373-5385).


J. H. Wicks, Manager
West Tank Farm Operations



Westinghouse
Hanford Company

Internal
Memo

From: Organic Tank Safety
Phone: 373-2238 R2-78
Date: February 28, 1994
Subject: TANK T-111

7A700-94.004

To: J. H. Wicks

cc: H. Babad	R2-78	J. W. Lentsch	R2-78
D. C. Board	S1-57	D. M. Lucoff	N1-36
V. C. Boyles	R1-49	G. J. Miskho	R2-12
D. A. Bragg	R1-49	D. J. Newland	R2-36
R. G. Brown	R2-11	R. Ni	S5-07
R. J. Cash	R2-78	J. W. Osborne	R2-78
G. M. Christensen	H4-21	M. A. Payne	R2-31
C. DeFigh-Price	R2-31	M. Plys	H2-62
A. J. Duckett	H0-30	A. Postma	H4-61
G. T. Dukelow	R2-78	T. E. Rainey	R4-02
J. C. Fulton	R2-31	R. E. Raymond	R2-54
J. M. Grigsby	H2-62	J. P. Sederburg	R2-11
G. A. Hanson	R2-08	M. H. Shannon	H4-61
J. O. Honeyman	R2-52	M. J. Sutey	R1-49
M. N. Islam	R2-08	O. S. Wang	R2-78
G. D. Johnson	R2-78	J. H. Wicks	T4-07
N. W. Kirch	R2-11	R. D. Wojtasek	R2-36
J. L. Lee	R2-36	DAT File/LB	

- References:
- (1) Internal Memo, D. A. Turner to H. Babad, et al., "Reactive Component in Tank 241-T-111," dated January 17, 1994.
 - (2) PNL Memo, D. L. Baldwin to R. M. Bean, et al., "T-111 Dry/Wet Preliminary TOC Results from Two Methods," dated January 11, 1994.
 - (3) Letter, S. G. McKinley, PNL, to C. DeFigh-Price, WHC, "Tank T-111, Core 33, Segment 2 Preliminary Data," dated December 20, 1993.
 - (4) Internal Memo, A. J. Duckett to D. B. Bechtold, et al., "Tank Farms PRC Meeting Minutes for December 1, 1993 - Potential Reactive Component in Tank 241-T-111," dated December 2, 1993.
 - (5) WHC-EP-0681, UC-600, "Interim Criteria for Organic Watch List Tanks at the Hanford Site," dated September 1993.

J. H. Wicks, et al.

7A700-94.004

Page 2

February 28, 1994

Waste Tank Safety Program recommends that tank T-111 be placed on the organic tanks Watch List, effective immediately.

The Tank Farms Plan Review Committee (PRC) made a decision on December 1, 1993, that tank T-111 was to be treated as an organics Watch List tank (Reference 4). This decision was prompted by the fact that portions of tank T-111 core material retrieved in 1991 and analyzed in 1992 exhibited significant exothermic activity in Differential Scanning Calorimetry (DSC) tests. The PRC also recommended that additional analyses be performed on existing samples (Reference 4).

PNL has rerun a number of key analyses on tank T-111 Core 33, Segment 2 samples (References 2 and 3). Reference 1 was issued after review of the PNL analytical results to put the tank T-111 issue into perspective, based on what we know at this time. Two conclusions were reached in Reference 1:

- No imminent safety hazard exists relative to the reactive component identified in tank T-111 waste because of the high (~80 wt.%) moisture content of the waste.
- Additional information on the active component in tank T-111 waste is required. The observed exothermic activity can not be fully accounted for based on reported total organic carbon (TOC) levels.

The recommendation to place tank T-111 on the organics tank Watch List at this time is based on the following rationale:

- Tank T-111 Core 33, Segment 2 sample analyses performed by PNL were conducted on material dried at 60°C under vacuum in an effort to reduce the scatter previously encountered in analytical data.
- PNL analytical results indicate exothermic activity in the dried sample of 215 cal/g (dry basis) from DSC tests (Reference 3). TOC content was determined to be 4.1 wt.% (dry basis) using the Furnace Total Combustion Method on dried sample material (Reference 2). Waste moisture content was confirmed to be approximately 80 wt.% (Reference 3).
- The attachment derives the energy equivalent of a 5 wt.% TOC (dry basis) organic-nitrate/nitrite mixture based on sodium acetate as the organic waste surrogate. 5 wt.% TOC (dry basis) is shown to be equivalent to exothermic activity of 151 cal/g.
- Reference 5 establishes interim criteria for organic Watch List tanks at the Hanford Site. Single-shell tank (SST) waste is to be classified as "conditionally safe" if its organic content exceeds 5 wt.% TOC (dry basis) and its moisture content is ≥ 17 wt. %.

J. H. Wicks, et al.
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February 28, 1994

7A700-94.004

- If, as shown above, 151 cal/g represents the energy equivalent of a 5 wt.% TOC (dry basis) organic-nitrate/nitrite mixture, then the waste in tank T-111 would be classified as "conditionally safe" in view of its 215 cal/g exotherm and 80 wt.% moisture content.
- SSTs with waste classified as "conditionally safe" are to be placed on the organic tanks Watch List (Reference 5).
- Tank T-111 should, therefore, be placed on the organic tanks Watch List.
- It should be noted that PNL's analytical results indicate a TOC content of only 4.1 wt.% (dry basis) (Reference 2). This TOC content, by itself, would not qualify tank T-111 for the Watch List. However, what matters from the standpoint of continued safe interim storage (e.g. safety) is waste energy content (cal/g). Tank T-111 clearly qualifies for the Watch List on this basis.

It appears that tank B-202 exhibits similar characteristics to those of tank T-111. However, it is not the recommendation of the Waste Tank Safety Program to place tank B-202 on the organic tanks Watch List at this time. The preliminary and incomplete analytical data have yet to be confirmed and further evaluation is necessary before making a final decision about tank B-202.

Please call (373-2238) if additional information or discussion would be beneficial.



D. A. Turner, Manager
Organic Tank Safety

mjs

Attachment

Modeling T-111

A simple model of T-111 was made on the ESP simulation program to attempt to shed some light on the vapor concentration issue. This simple model would mix about 1 liter of aqueous solution similar to T-111 and 1 liter of air. Some NPH type organics were also mixed. The vapor phase leaving the mixture was estimated.

Aqueous solution

The aqueous solution used for this simulation was:

H2O	1000 g
NaNO2	0.54 g
NaNO3	30.7 g
Na2CO3	0.5 g

The model calculated this to be 1.017 liters with a density of 1.026 compared with T-111 liquid density of 1.07.

AIR

One liter of air was introduced assuming only O2 and N2.

The model calculated that there was 1.08 liters of air

Organic

The organic chosen was of the NPH type.

C10H22	0.3 g
C11H24	0.15 g
C12H26	0.05 g

This mixture is just an estimation.

Mixture Conditions

The mixture conditions were 72 °F and 14.7 psia.

Case #1

A total of 0.5 grams of the organic mixture was added to the mixture.

This resulted in the following vapor composition:

Component	Vol %
H2O	2.62 %
O2	18.81%
N2	71.68%
C10H22	4.32%
C11H24	1.97%
C12H26	0.60%

The remainder of the organic was soluble in the liter of aqueous solution. There was no organic phase.

Note that the vapor phase contained large amounts of the hydrocarbons. This is well above what was measured. Several orders of magnitude.

Conclusions:

T-111 does not need a separable phase organic layer to explain the observed vapor pressure of 9 ppm. Dissolved organics in an aqueous phase can explain this.

Had T-111 had a small amount of NPH floating on it, it probably would have evaporated by now. Notice that the C10H22 is very much greater in concentration. This means that over time, the C12H26 would tend to be left behind. This has a smaller vapor pressure and is safer.

Distribution

LR Pederson	K2-44
MG Plys	H4-62
LL Burger	P7-25
RD Scheele	P7-25
GL Borsheim	R2-11
DM Camaioni	K2-38
MH Campbell	R3-77
BC Simpson	R2-12
WD Samuels	K2-44
JA Campbell	P8-08
JP Sederberg	R2-11
H Babad	R2-78



Pacific Northwest Laboratories

RECEIVED

Project Number _____

Internal Distribution _____

JAN 20 1994

R.M. BEAN

Date January 14, 1994

To - RM Bean

From DL Baldwin *DL Baldwin*Subject FINAL T-111 (Core 33. Segment 2) DRY/AS-
RECEIVED TOC RESULTS FROM TWO METHODS. Rev. 2.

JM Tingey
MW Urie
RT Steele
SG McKinley
EW Hoppe
File/LB

This is the Final report of results from TOC/TIC/TC measurements of the analyzed "dried" and "as-received" T-111 sample, ALO No. 94-2631, analyzed by two methods, as follows:

- 1) Hot Persulfate method, in normal TIC/TOC mode, and TC mode,
- 2) Furnace Total Combustion method, in both TOC mode and TC mode.

The data on the drying of the sample, as performed by JM Tingey, shows the sample to contain 18.4% solids. In this report, no corrections are made for the "dried" samples results.

Description of Methods: Part 1 of this work is done by the hot persulfate oxidation method, Test Procedure PNL-ALO-381, rev. 0, "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils and Sludges by Hot Persulfate Method". The M&TE No. for the carbon measurements is WC01713, the balance M&TE No. is 360-06-01-016. The data is located on the accompanying data sheets, review reports or on file in the ALO Records Office. TOC standard used is alpha-d-glucose, Kodak lot# B1F, and the TIC standard is reagent-grade Na_2CO_3 . Both materials are used in liquid form, dissolved in water, for system standards as well as matrix spikes. Normal operation is with a TIC step first, using acid only, followed by the TOC step on the same sample, adding the persulfate. TC is calculated by addition. A modified mode was used in which TC was determined, eliminating the TIC step. This should minimize possible loss of any unmeasured volatile organics. The estimated precision is $\pm 10\%$ and the estimated accuracy is $\pm 15\%$.

Part 2 of this work is done by the furnace total combustion method, Test Procedure PNL-ALO-380, rev. 0, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer". The M&TE No. for the carbon measurements is WD04981, the balance M&TE No. is 360-06-01-023. The data is located on the accompanying data sheets, review reports or on file in the ALO Records Office. TOC standard used is alpha-d-glucose and the TIC standard is CaCO_3 . Both materials are used in solid form for system standards as well as matrix spikes. As per normal procedure, TOC is determined at 600°C on a sample. Then TC is determined at 1000°C on a different sample. TIC is calculated by difference. The estimated precision is $\pm 10\%$ and the estimated accuracy is $\pm 15\%$.

QC Narrative: This sample was analyzed on 1-10-94. The QC for both methods all came within required MCS-033 limits, with no apparent outliers. For the

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persulfate method, the TIC and TOC system standard recoveries ranged from 91.9% to 101.5%. The system blanks were consistent. The RPD's (Relative Percent Difference) for the dried sample, analyzed in duplicate, ranged from 3% to 10%. The matrix spike recoveries were 95% and 119%, respectively, for TIC and TOC, and 119% for the "crushed" sample TC spike.

For the furnace total combustion method, the TIC and TOC system standard recoveries ranged from 93.9% to 99.3%. The system blanks were consistent. The RPD's for the dried and as-received samples, analyzed in duplicate, ranged from 3% to 16%. The matrix spike recoveries, at 600°C, were 102% and 83%, respectively, for the dried and as-received samples.

Table 1: Hot Persulfate Method Results

Sample	sample wt (g)	TIC (%)	RPD (%)	TOC (%)	RPD (%)	TC (%)	RPD (%)
T-111 Dry-1	0.1304	0.41		0.94		1.35	
T-111 Dry-2	0.1857	0.42	3	1.04	10	1.46	8
				(avg=0.99%)		(avg=1.41%)	
T-111 Dry-3 Spike		95% TIC Recovery			119% TOC Recovery		
T-111 Dry-4 (crushed)	0.1638	0.52		1.74		2.26	
T-111 Dry-A (crushed)						1.90	
T-111 Dry-B (crushed)						2.09	10
				(avg=2.00%)			
T-111 Dry-C (crushed) Spike					119% TC Recovery		

- Notes:
- 1) Undissolved small black chunks of material were observed in reaction flask, indicating incomplete reaction.
 - 2) The dried material was composed of large chunks, therefore it was crushed, in order to allow better dissolution. Improved dissolution was observed, but still black undissolved fines were seen.
 - 3) Samples 1 through 4 were analyzed as per normal TIC/TOC procedure. Sample A through C were analyzed by modified TC procedure. In this case, the persulfate and acid were added at the same time to the sample, deleting the TIC step, minimizing possibility of loss of volatile organic material.

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Table 2: Furnace Method Results

Sample	sample wt (g)	TIC (%)	RPD (%)	TOC (%)	RPD (%)	TC (%)	RPD (%)
T-111 Dry-1 (600°C)	0.01495			3.76			
T-111 Dry-2	0.01864			4.41	16		
T-111 Dry-3, Spike	0.02345	102% Recovery		(avg=4.09%)			
T-111 Dry-1 (1000°C)	0.03158					4.69	
T-111 Dry-2	0.02333					4.99	6.2
						(avg=4.84%)	
T-111 As-Recd-1 (600°C)	0.04890			0.90			
T-111 As-Recd-2	0.10883			0.87	3.4		
T-111 As-Recd-3, Spike	0.09272	83% Recovery		(avg=0.89%)			
T-111 As-Recd-1 (1000°C)	0.04227					0.98	

Note: 1) In the furnace method, TOC only is considered to be released at 600°C. At 1000°C, TC is considered to be released. Note that different weighed samples are used at the two different temperatures.

CONCLUSIONS:

- 1) The Hot Persulfate method alone provides one conclusion, that there is no loss of volatile organic material in the TIC step. The results appear to agree well with the earlier reported "as-received" sample results, after accounting for the water weight loss.
- 2) The Furnace method results, in comparison, indicate that there is additional organic material present in these samples. This additional organic material was, for some reason, not fully oxidized by hot persulfate, but required oxidizing with some stronger oxidant, (e.i., oxygen) at elevated temperatures.
- 3) In the dry material, from the Furnace method, the total TOC found was 4.09%, the total TC found was 4.84%. This compares, from the hot persulfate method, with total TOC found of 0.99% and total TC found

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of 2.0%. Both method results show good agreement between "dried" and -
"As-Recieved" results, after accounting for water weight loss.

- 4) The missing, additional organic material, in the dried sample, amounts to:

TC comparison: $(4.84\% - 2.00\%) = 2.8\%$
TOC comparison $(4.09\% - 0.99\%) = 3.1\%$

Concur by: McBurt
File: T-111.RMB
System File: T0C11094

Date: 1/14/94

DON'T SAY IT --- Write It!

DATE: June 9, 1994

TO: Distribution

FROM: C. H. Delegard

Telephone: 373-4658

SUBJECT: CENTIFUGATION AND ANALYSES OF SLUDGE FROM TANK 241-T-111

Attached please find Internal Memo 8E110-PCL94-046,
"Centrifugation and Analyses of Sludge From Tank 241-T-111."
The attached IM provides recent thermal analyses of the
centrifuged sludge and completes the reporting of the
tank 111-T centrifugation tests. The attached IM replaces
IM 8E110-PCL94-043, same subject. Please discard the IM
8E110-PCL94-043.

**Westinghouse
Hanford Company****Internal
Memo**

From: Process Chemistry Laboratories 8E110-PCL94-046
Phone: 373-4658 T6-09
Date: June 9, 1994
Subject: CENTRIFUGATION AND ANALYSIS OF SLUDGE FROM TANK 241-T-111

To: D. B. Engelman : -49

cc: H. Babad R2-78
G. S. Barney T5-12
D. R. Bratzel R2-12
J. M. Frye T6-30
J. R. Jewett T6-09
J. G. Kristofzki T6-06
L. M. Sasaki R2-12
C. S. Simmons K6-77
B. C. Simpson R2-12
J. P. Slougher T6-07
C. S. Sutter T5-12
D. T. Vladimiroff R2-12
CHD File/LB

References:

- (1) Internal Memo, G. S. Barney and C. H. Delegard to D. B. Engelman; "Test Plan for Centrifuge Drainage Tests of Tank 241-T-111 Sludge Samples," dated May 3, 1994.
- (2) Internal Memo, G. S. Barney to D. W. Jeppson, "Results of Centrifuge Drainage Tests for Simulated Infarm-2 Ferrocyanide Sludge," dated March 28, 1994.

INTRODUCTION AND SUMMARY OF RESULTS

In Reference 1, a laboratory test procedure was described to determine the liquid retention capacity of sludge from tank 241-T-111 (111-T). The test procedure was based on a study, described in Reference 2, conducted to measure sludge drainage rates. In the present studies, tests were designed to simulate the drainage of interstitial liquid from the existing 14-foot (4.3 meter) overburden of sludge in 111-T should salt well pumping at the tank bottom take place.

Pressure-enhanced drainage was imposed in the laboratory by removing interstitial solution from samples of genuine sludge by filtration through coarse-frit glass in a centrifugal field of 500 times gravity (500 G). This field simulates the 4.3-meter sludge hydrostatic pressure at the tank bottom. The weights of liquids extracted and sludge retained were measured intermittently over a period of about five days. By knowing the water concentration of the original sludge and the expressed liquid, the amount of liquid remaining in the sludge cake could be determined and compared with

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water contents of the sludge cake measured directly at the end of centrifugation.

Four sludge samples from 111-T were tested in duplicate by this procedure. Results showed the water content of the initial sludges ranged from about 69 to 80 weight percent (wt%). Centrifugation filtering produced compressed, but still damp, sludge materials and quantities of expressed liquid. The rate of liquid removal from the sludge cake was rapid in the initial hours of centrifugation and slowed considerably after two days' centrifugation. The water content of the sludges after two days' centrifuging ranged from 58 to 66 wt%; after five days, the water content decreased a further 1.6 wt% for each sludge on average.

The experiments and measurements performed to determine the solution retention of the centrifuged sludge samples are described in this report. Ancillary chemical and thermal analyses of the intact and separated sludge and solution fractions also are reported.

EXPERIMENTAL MATERIALS AND METHODS

Archive sludge samples taken in 1991 were used in these tests. The sample materials were derived from segments 3 and 7 of core 31 and from segments 1 and 7 from core 33. The segment numbering proceeds, in order, from top (segment 1) to bottom (segment 9) in 19-inch (0.5-m) increments corresponding to the length of the segments.

The experimental approach used in the centrifugation tests generally followed the methods described in reference 1. The experimental observations were recorded in notebook WHC N 656 1

Coarse-frit glass filters (40 to 60 μm pore size) fused into 1-cm diameter glass tubes (about 8 cm long) were used as the filtration medium. Samples of well-mixed sludge were introduced to the top of frit filter surface by plunger-type polyethylene thief samplers. Tare and gross weight measurements (± 0.00005 grams using a five-place balance) of the tubes before and after introduction of sample were obtained. The glass tubes were placed in screw-top polypropylene 50-mL capacity centrifuge tubes and reweighed. Sample weights ranged from about 1.8 to 3.4 grams. Most sample weights were about 2.3 grams.

The tubes with samples were spun in a centrifuge located in an open-face hood in the 222-S Laboratory at a target velocity of 1680 revolutions per minute (RPM). This velocity corresponded, at the 15.8 cm distance of the sludge from the axis of rotation, to a field of 500 G at the filter frit disc. Measurements of the rotational velocity of the centrifuge were obtained during the course of the experimentation using an optical tachometer. The nine velocity measurements ranged from 1617 to 1720 RPM (i.e., 464 to 524 G) and averaged 1684 RPM (501 G). The temperature within the centrifuge was about 1°C above ambient lab temperature. The lab temperature ranged from about 19 to 24°C during the experiments.

The centrifuge was stopped periodically, the glass tubes and centrifuge cones were weighed and the weights of centrifuged liquid and sludge were determined by difference. The liquid was sampled for analyses of water content, density, thermal behavior and concentration of total organic carbon (TOC). After 113 hours, the centrifugation was stopped and the sludge analyzed for water content and thermal behavior. Samples of the original sludge also were analyzed for water content and thermal behavior. Sludge water content was determined by weight loss on oven heating to constant weight at 116°C. For the water content determinations, original sludge material sample sizes were 1 to 2 g; about 0.2 to 0.5 g samples were used for the centrifuged sludge materials.

Densities of the original and centrifuged sludge were determined. The densities of original sludge were determined by weighing sludge samples into volume-calibrated graduated centrifuge tubes and noting the total sludge solution plus settled solids volume after centrifugation.

The compressed centrifuged sludges from the solution retention tests were not sufficiently plastic to slump in the volumetric centrifuge tubes used for density measurement. Therefore, the densities of the compressed sludge were determined via a displacement technique using an immiscible liquid of known density (*n*-hexane). Samples of the compressed sludge were added to a tare-weighed 5-mL volumetric flask, the flask reweighed, the flask filled to the mark with *n*-hexane, and the flask again reweighed. The volume of added *n*-hexane could be determined by weight and density. The volume and weight of compressed sludge then was calculated by difference and the sludge density calculated.

All chemical, physical and thermal analyses (except the water concentrations of the centrifuged sludges and the densities of the original and centrifuged sludges) were determined by 222-S Analytical Operations. The densities of the expressed liquids were determined by weighing known volumes of solution held in volume-calibrated pipets. The TOC concentrations were found by acidified sample pyrolysis followed by coulometric titration of the product carbon dioxide collected in a scrubber trap. Water content in the expressed solution was determined by weight loss of sample due to heating in an oven.

The thermal analyses included differential scanning calorimetry (DSC) and thermogravimetric analyses (TGA). All sludge and expressed solutions were analyzed by DSC/TGA. The DSC determined if a heat-producing (exothermic) event occurred. An example of an exothermic reaction would be the oxidation of organic carbon by nitrate. The TGA showed fractional weight changes (e.g., weight loss by water evaporation) as a function of increasing temperature. Thus water concentration values were obtained via TGA for all samples.

RESULTS AND DISCUSSION

All the original sludge samples were dark brown in color with a pasty but slightly gritty consistency. Sludge samples from the segment 1 and 3 archives showed no natural segregation whereas the segment 7 samples had about 10% supernatant solution. The entire contents of each archive sample

were mixed prior to drawing subsamples for testing. Subsampling from the same archive vials two days later showed separation of supernatant solution had recurred in the segment 7 samples but again no supernatant solution was found for the segment 1 and 3 samples. Centrifugation resulted in the drainage (through the glass frit filters) of yellow liquids from all sludge samples and the production of a sludge cake that was smaller both in height and diameter than the original sludge (i.e., the sludge compressed as well as shrunk away from the glass tube wall).

The water content of the centrifuged sludge was calculated by weight loss of the sludge as centrifugation proceeded, the water content of the original sludge, and the water content of the expressed liquid. The water content of the centrifuged sludge, after 113 hours' centrifugation at 500 G, was also determined directly via oven weight loss. The water content data, presented in the Figure, show the original sludge was about 80 wt% water for the segment 1 and 3 samples and about 74 wt% for the segment 7 samples taken near the tank bottom. Though the archive segment 7 samples had supernatant liquid while the segment 1 and 3 samples did not, the segment 7 samples had lower water content than the segment 1 and 3 samples.

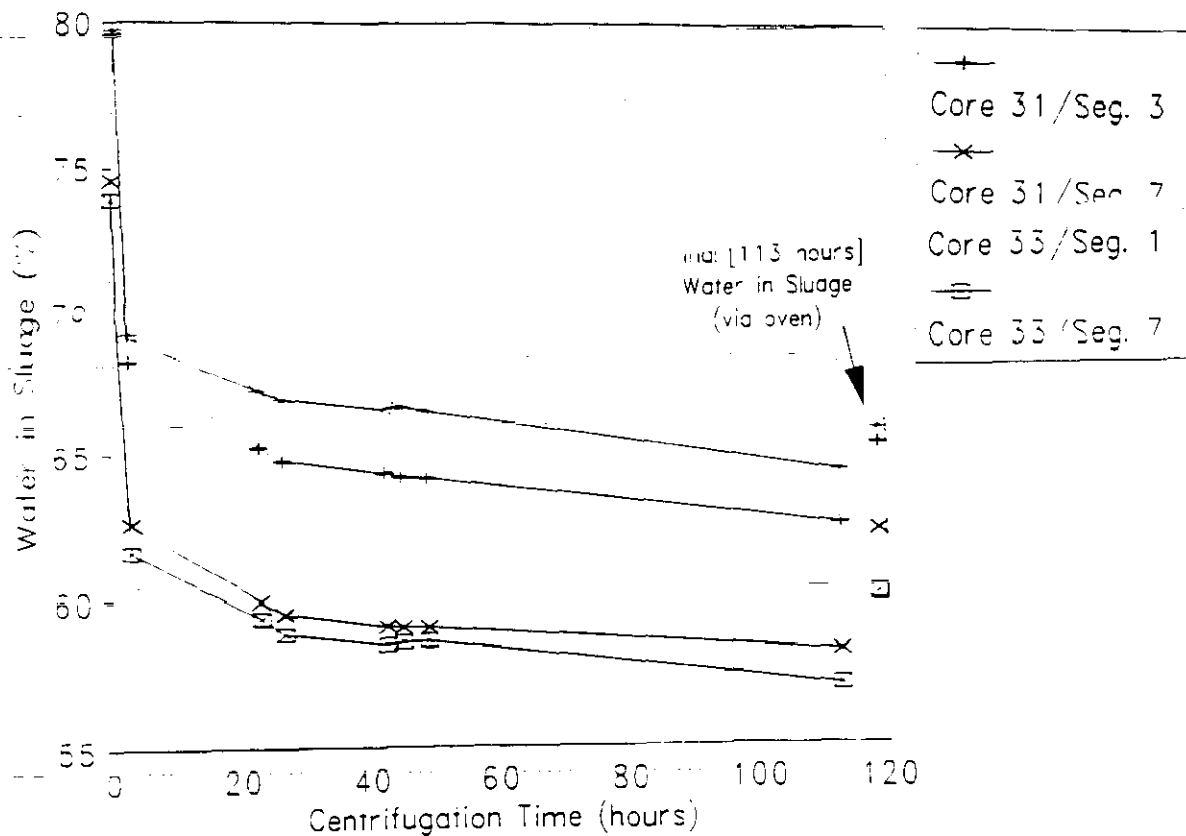


Figure. Water Concentration in Centrifuged 111-T Sludge.

The sludge samples lost solution (and water content) quickly in the initial three hours of centrifugation. The rate of weight loss decreased with time of centrifugation. After 113 hours, the water contents of the segment 1 and 3 samples were about 63 wt%; the segment 7 samples were about 58 wt%. The water contents of the centrifuged sludges determined by direct measurement of oven weight loss were about 65 wt% and 61 wt%, respectively.

The water content data of the individual replicate sludge samples are given in Table 1. The data show precision in the replicate measurements of about 0.5% relative or 0.8% absolute in the oven measurements and about 1.3% relative or 1.9% absolute for the calculated values based on the sludge weight losses. The oven values are about $2.8 \pm 1.0\%$ higher, on average, than the calculated values. The bias probably is due to the cumulative effects of water vapor loss from the centrifuged solution during each weighing operation. For this reason, the oven wt% water values are judged to be more reliable than the calculated values.

Table 1. Water Content of Sludge During Centrifugation.

Core/ Seg/ Dup	Centrifugation Time (hours)								
	0	3	23	26.5	42.5	45	49	113	113 (Oven)
31/3/1	79.54	68.6	65.8	65.4	64.7	64.5	64.6	63.0	65.14
31/3/2		67.7	64.4	63.9	63.6	63.5	63.4	61.5	64.78
31/7/1	74.52	63.1	60.6	60.2	59.7	59.8	59.8	59.1	62.28
31/7/2		62.0	59.2	58.7	58.3	58.2	58.2	57.1	61.83
33/1/1	79.71	68.7	67.1	66.8	66.4	66.5	66.3	64.4	65.74
33/1/2		69.3	67.0	66.7	66.3	66.4	66.2	63.8	65.23
33/7/1	73.87	61.8	59.9	59.4	59.1	59.2	59.3	57.3	59.66
33/7/2		61.3	58.8	58.2	57.7	57.8	57.8	56.6	60.23

The chemical and thermal analyses of the original sludge materials are summarized in Table 2. Exotherms were found for all sludge samples. The exotherms decreased as the sludge sampling depth increased (i.e., exotherms were greatest at the top). The wt% water values of the original sludge materials were determined both by weight loss, using 1 to 2 g samples heated to dryness in an oven, and by TGA, using 30 to 50 mg samples.

As shown in Table 2, the oven values for wt% water were higher than the TGA values. The values derived by oven weight loss are judged to be more reliable than the TGA values because they showed higher precision (better reproducibility in their duplicates), were obtained from larger, more representative samples, and thus were less subject to evaporative weight losses prior to initial weighing. The smaller TGA samples would be apt to lose more weight, on a relative basis, than the larger oven samples because they have a higher surface area to volume ratio. The oven-determined wt% water duplicate values for particular core/segment samples were averaged for

use in calculation of the water content of the corresponding centrifuged sludge.

Table 2. Analyses of the Original Sludge.

Core/Seg/Dup	Wt% Water		Density (g/mL)	Exotherm (J/g)
	Oven	TGA		
31/3/1	79.47	76.33	1.24	191
31/3/2	79.60	77.10		112
31/7/1	74.28	72.99	1.19	33.1
31/7/2	75.16	75.12		10.2
33/1/1	79.64	77.12	1.16	249
33/1/2	79.48	79.04		254
33/7/1	73.93	81.95	1.20	41.4
33/7/2	74.20	74.24		37.5

The chemical, physical and thermal analyses of the solution samples are summarized in Table 3. Though the samples (especially the segment 1 and 3 materials) contained organic carbon, no exotherm was noted in the DSC of any of the solutions. Again, both oven and TGA values of wt% water were determined and, for the same reasons, the oven values judged to be more reliable. The average value of the oven-determined wt% water duplicate analyses for particular core/segment samples was used to calculate the water content of the corresponding centrifuged sludge.

Table 3. Analyses of Expressed Solution.

Core/Seg/Dup	Wt% Water		Density (g/mL)	[TOC] (mg/L)
	Oven	TGA		
31/3/1	92.27	86.80	Insuff. sample	1620
31/3/2	91.65	85.13	1.040	979
31/7/1	88.87	80.80	1.065	210
31/7/2	88.52	85.40	1.087	140
33/1/1	95.41	87.02	0.990	1010
33/1/2	94.65	92.06	1.028	1000
33/7/1	90.02	85.50	1.089	180
33/7/2	89.44	86.66	1.101	200

The chemical and physical analyses of the centrifuged sludge samples are given in Table 4. The wt% water derived from the TGA are significantly lower than those found by oven drying. Substantial air-drying of the centrifuged sludge apparently occurred prior to the TGA determinations. The DSC analyses showed that on a dry basis, centrifuged sludges had exotherms somewhat larger than the original sludges. For example, for sludge from

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segment 1 of core 33 (which exhibited the highest exotherm of the four samples studied), the centrifuged material had an exotherm of about 1700 J/g dry sludge as compared to 1140 J/g dry sludge for the original material. For segment 3 of core 31, the centrifuged sludge gave about 1150 J/g dry sludge (the 1525 J/g value was not included in the calculation) while the original sludge gave about 640 J/g dry sludge. The centrifuged segment 7 samples showed no measurable exotherms; exotherms for the original segment 7 sludges were about 100 to 200 J/g dry sludge.

Table 4. Analyses of Centrifuged Sludge.

Core/Seg/Dup	Wt% Water		Density (g/mL)	Exotherm (J/g)
	Oven	TGA		
31/3/1	65.14	53.80	1.05	546.9, 465.3
31/3/2	64.78	56.92	1.12	1525.3, 544.9
31/7/1	62.28	58.94	1.16	0, 0
31/7/2	61.83	52.71	1.24	0
33/1/1	65.74	51.11	1.21	838.1
33/1/2	65.23	51.62	1.16	822.4
33/7/1	59.66	44.82	1.24	0
33/7/2	60.23	45.48	1.33	0

Please call should you have questions or comments on this work.



C. H. Delegard, Principle Senior Scientist
Process Chemistry Laboratories

am

SAMPLE STATUS REPORT FOR T 546. T-111 T-111 #1 TIME: 3/ 9/94 9:31
DISPATCHED: 3/ 5/94 10:31 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 3/ 5/94 13:15

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	*****	*****	***	***	*****
1001	APPR/OTR	YELLOW CLEAR < 1% SOLIDS			N54D2
5706	SPG	1.03610E 00 NONE	N	Y	N54D2
5711	DSC	OUT FOR RERUN			N54D2
5711	DSC	INCOMPLETE			N54D2
5712	TGA	INCOMPLETE			N54D2
5713	pH	1.15700E 01 NONE	N	Y	N54D2
5714	% H2O	OUT FOR RERUN			N54D2
5714	% H2O	INCOMPLETE			N54D2
5720	TB	2.21000E-01 uCi/ML	N	Y	N54D2
5725	AT	2.31000E-03 uCi/ML	N	Y	N54D2
5726	TOC	4.73000E 02 uG C/ML	N	Y	N54D2
5727	TIC	8.00000E 02 uG C/ML	N	Y	N54D2
5728	NH4	3.61000E 02 uG/ML	N	Y	N54D2
5729	OH LIQ	3.54000E 03 uG/ML	N	Y	N54D2
5730	GEA	8.96000E-02 uCi/ML	N	Y	N54D2
5750	ICP-LIQ	2.04000E 04 uG/ML	N	Y	N54D2
5750	ICP-LIQ	< 5.05000E 00 uG/ML	N	Y	N54D2
5771	IC	1.95997E 03 uG/ML	N	Y	N54D2
5771	IC	4.77260E 02 uG/ML	N	Y	N54D2
5771	IC	1.33501E 03 uG/ML	N	Y	N54D2
5771	IC	2.91022E 04 uG/ML	N	Y	N54D2
5771	IC	8.06644E 03 uG/ML	N	Y	N54D2
5771	IC	2.78205E 03 uG/ML	N	Y	N54D2
5778	CN LIQ	2.71000E 00 uG/ML	N	Y	N54D2
5781	Pu239/40	OUT FOR RERUN			N54D2
5781	Pu239/40	INCOMPLETE			N54D2
5782	Am241	< 3.97000E-05 uCi/ML	N	Y	N54D2
5783	Np237	< 1.38000E-05 uCi/ML	N	Y	N54D2
5786	Sr90	OUT FOR RERUN			N54D2
5786	Sr90	7.09000E-04 uCi/ML	N	Y	N54D2
9114	ARCHIVE	INCOMPLETE			N54D2

END OF REPORT

SAMPLE STATUS REPORT FOR T 548. T-111 T-111 #2 TIME: 3/ 9/94 9:32
DISPATCHED: 3/ 5/94 10:48 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 3/ 5/94 13:16

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	*****	*****	***	***	*****
1001	APPR/OTR	CLEAR YELLOW < 1% SOLIDS			N54D2
5706	SPG	1.03760E 00 NONE	N	Y	N54D2
5711	DSC	OUT FOR RERUN			N54D2
5711	DSC	INCOMPLETE			N54D2
5712	TGA	INCOMPLETE			N54D2
5713	pH	1.15900E 01 NONE	N	Y	N54D2
5714	% H2O	9.29000E 01 % H2O	N	Y	N54D2
5720	TB	2.31000E-01 uCi/ML	N	Y	N54D2
5725	AT	2.32000E-03 uCi/ML	N	Y	N54D2
5726	TOC	4.18000E 02 uG C/ML	N	Y	N54D2
5727	TIC	7.90000E 02 uG C/ML	N	Y	N54D2
5728	NH4	3.70000E 02 uG/ML	N	Y	N54D2
5729	OH LIQ	2.70000E 03 uG/ML	N	Y	N54D2
5730	GEA	9.21999E-02 uCi/ML	N	Y	N54D2
5750	ICP-LIQ	2.43000E 04 uG/ML	N	Y	N54D2
5750	ICP-LIQ	< 5.05000E 00 uG/ML	N	Y	N54D2
5771	IC	2.15977E 03 uG/ML	N	Y	N54D2
5771	IC	4.97640E 02 uG/ML	N	Y	N54D2
5771	IC	1.37823E 03 uG/ML	N	Y	N54D2
5771	IC	3.00138E 04 uG/ML	N	Y	N54D2
5771	IC	8.24791E 03 uG/ML	N	Y	N54D2
5771	IC	2.85180E 03 uG/ML	N	Y	N54D2
5778	CN LIQ	1.84000E 00 uG/ML	N	Y	N54D2
5781	Pu239/40	OUT FOR RERUN			N54D2
5781	Pu239/40	INCOMPLETE			N54D2
5782	Am241	< 2.76000E-05 uCi/ML	N	Y	N54D2
5783	Np237	< 2.89000E-05 uCi/ML	N	Y	N54D2
5786	Sr90	OUT FOR RERUN			N54D2
5786	Sr90	3.03000E-04 uCi/ML	N	Y	N54D2
9114	ARCHIVE	INCOMPLETE			N54D2

END OF REPORT

SAMPLE STATUS REPORT FOR T 550. T-111 T-111 #3 TIME: 3/ 9/94 9:32
 DISPATCHED: 3/ 5/94 10:49 SAMPLE HAS NOT BEEN SLURPED
 RECEIVED: 3/ 5/94 13:16

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	*****	*****	***	***	*****
1001	APPR/OTR	CLEAR YELLOW < 1% SOLIDS			N54D2
5706	SPG	1.03310E 00 NONE	N	Y	N54D2
5711	DSC	OUT FOR RERUN			N54D2
5711	DSC	INCOMPLETE			N54D2
5712	TGA	INCOMPLETE			N54D2
5713	pH	1.17800E 01 NONE	N	Y	N54D2
5714	% H2O	9.29000E 01 % H2O	N	Y	N54D2
5720	TB	2.48000E-01 uCi/ML	N	Y	N54D2
5725	AT	2.49000E-03 uCi/ML	N	Y	N54D2
5726	TOC	3.80000E 02 uG C/ML	N	Y	N54D2
5727	TIC	OUT FOR RERUN			N54D2
5727	TIC	4.07000E 02 uG C/ML	N	Y	N54D2
5728	NH4	5.22000E 02 uG/ML	N	Y	N54D2
5729	OH LIQ	2.89000E 03 uG/ML	N	Y	N54D2
5730	GEA	8.78000E-02 uCi/ML	N	Y	N54D2
5750	ICP-LIQ	2.60000E 04 uG/ML	N	Y	N54D2
5750	ICP-LIQ	< 5.05000E 00 uG/ML	N	Y	N54D2
5771	IC	2.18774E 03 uG/ML	N	Y	N54D2
5771	IC	5.11930E 02 uG/ML	N	Y	N54D2
5771	IC	1.40658E 03 uG/ML	N	Y	N54D2
5771	IC	3.16720E 04 uG/ML	N	Y	N54D2
5771	IC	8.83930E 03 uG/ML	N	Y	N54D2
5771	IC	3.14581E 03 uG/ML	N	Y	N54D2
5778	CN LIQ	2.39000E 00 uG/ML	N	Y	N54D2
5781	Pu239/40	OUT FOR RERUN			N54D2
5781	Pu239/40	INCOMPLETE			N54D2
5782	Am241	< 2.81000E-05 uCi/ML	N	Y	N54D2
5783	Np237	< 2.68000E-05 uCi/ML	N	Y	N54D2
5786	Sr90	OUT FOR RERUN			N54D2
5786	Sr90	1.21000E-03 uCi/ML	N	Y	N54D2
9114	ARCHIVE	INCOMPLETE			N54D2

END OF REPORT

Estimate of plutonium concentration that would place 241-T-111 out of specification with regard to established inventory limits.

50 kg $^{239/240}\text{Pu}$ is the standard inventory limit for single-shell tanks¹.

$$\text{Therefore, } \frac{50,000 \text{ g } ^{239/240}\text{Pu}}{2.171\text{E}+09 \text{ g waste}} = \frac{2.303\text{E}-05 \text{ g } ^{239/240}\text{Pu}}{\text{g waste}}$$

Approximate proportion of Pu-239 = 96%

Approximate proportion of Pu-240 = 4%

Specific activity of Pu-239 = $6.2\text{E}-02 \text{ Ci/g}^2$

Specific activity of Pu-240 = $2.27\text{E}-01 \text{ Ci/g}$

Specific activity for the plutonium in the waste:

$$0.96*(6.2\text{E}-02 \text{ Ci/g}) + .04*(2.27\text{E}-01 \text{ Ci/g}) = 0.0686 \text{ Ci/g } ^{239/240}\text{Pu}$$

$$\frac{2.303\text{E}-05 \text{ g } ^{239/240}\text{Pu}}{\text{g waste}} * \frac{0.0686 \text{ Ci}}{\text{g } ^{239/240}\text{Pu}} * \frac{1\text{E}+06 \text{ } \mu\text{Ci}}{\text{Ci}} = \frac{1.58 \text{ } \mu\text{Ci}}{\text{g waste}}$$

The estimates of historical inventory values for selected analytes were derived from Agnew³. The estimates for 2C concentrations were done by taking the average concentrations of the two flowsheet formulations, 2C 44-51 and 2C 52-56. The 224 waste concentrations were used as presented. These values were multiplied by their respective volumes to develop the bulk inventory estimates. The bulk concentrations presented for the tank were derived by multiplying each analyte's concentration by the fraction its waste contributes to the total inventory and adding them together, i.e. $0.526*(2C_{\text{avg}} \text{ concentration}) + 0.474*(224 \text{ concentrations}) = \text{Bulk analyte concentration}$

¹Boyles, V. C. 1992, *Operating Specifications for Single-Shell Waste Storage Tanks*, OSD-T-151-00013, Rev. D-1, Westinghouse Hanford Company, Richland, Washington.

²Van Tuyl, H. H., 1962, *Fission Product Generation and Decay Calculations*, HW-75978, General Electric Company, Richland, Washington.

³Agnew, S. F. 1994, *Hanford Defined Wastes: Chemical and Radionuclide Compositions*, LA-UR-2657, Los Alamos National Laboratory, Los Alamos, New Mexico.

		MW 44- S1	MW 52- S6	1C44- S1/CW	1C52- S6/CW	2C44-51	2C52-56#224	UR/TBP	PFcCN1	PFcCN2	TFcCN	1CFcCN	R', 52- S6	R', 59- S67	CWR/AI, 50-61
frac. prec. solid/MW				1C44- S1/CW	1C52- S6/CW	2C44-51	2C52-56#224	UR/TBP	PFcCN1	PFcCN2	TFcCN	1CFcCN	R', 52- S6	R', 59- S67	CWR, 50-61
NaNO3		0	0	0	0	0	0	0.126	0.13546	0.13547	0	0	0	0.2805	0
NaNO2		0	0	0	0	0	0	0	0	0	0	0	0	0	0
NaCl		0	0	0	0	0	0	0	0	0	0	0	0	0	0
NaF		0	0	0.1706	0.1763	0.1058	0.3586	0.0634	0.06342	0.1639	0	0	0	0	0
Na2CO3		0.1	0.1	0	0	0	0	0	0	0	0	0	0	0	0
Na3PO4		0.26873	0.2842	0.5587	0.5891	0.3119	0.0047	0	0	0	0	0.583	0	0	0
Na2SO4		0.48274	0.4741	0	0	0	0	0.1475	0.35513	0.35514	0	0	0	0	0
Na2SiO3		0	0	0.5024	0.5058	0.4683	0.231	1	0.43804	0.43805	1	0.4983	1	1	0.3509
Al2O3.3H2O		0.6	0.6	0.8	0.9	0.3	0.6	0.07	0.07	0.07	0.07	0.07	0.6	0.07	0.2
NaAlO2		0	0	0	0	0	0	0	0	0	0	0	0	0	0
FeO(OH)		1	1	0.937	0.9374	0.918	0.8814	1	0.9337	0.85951	0.85951	1	0.9365	0.7404	0.8474
Cr(OH)3		1	1	0	0	0	0	1	1	1	1	1	0.5132	0.7165	1
MnO2		1	1	1	1	1	1	1	1	1	1	1	1	1	1
BPO4		1	1	0.8987	0.8994	0.8525	0.7866	0.7587	1	0.88653	0.88653	1	0.8979	1	1
Pb(OH)2		1	1	1	1	1	1	1	1	1	1	1	1	1	1
La2O3							0.6012								
Ce2O3							0.7								
Na3Cit.5H2O		0	0	0	0	0	0	0	0	0	0	0	0	0	0
Na Acetate		0	0	0	0	0	0	0	0	0	0	0	0	0	0
Na 2 Oxalate		0	0	0	0	0	0	0.25	0	0	0	0	0	0	0
Na3HEDTA		0	0	0	0	0	0	0	0	0	0	0	0	0	0
Na4EDTA		0	0	0	0	0	0	0	0	0	0	0	0	0	0
CaCO3.6H2O		1	1	1	1	1	1	1	1	1	1	1	1	1	1
Ni(OH)2		1	1	1	1	1	1	1	1	0.29194	0.84	0.6569	1	1	1
ZrO(OH)2		1	1	0.2909	0.2957	1	1	1	1	1	1	0.2851	1	1	1
Na2NiFe(CN)6.6H2O				1	1	1	1	1	1	1	1	1	1	1	1
Ru		0	0.5	0	0.536	0	0	0	0	0	0	0.5291	0	0.2823	0.8964
UO2(OH)2.6H2O		0.75	0.75	0.9413	0.9811	0.9508	0.9289	0	0.49	0.49568	0.49569	0.4872	0.9808	0.1873	0.5599
Cs		0	0	0	0	0	0	0.126	1	1	1	1	0	0.2805	0
Sr		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.1	0.6
pred. sludge ppm		MW		1C44- S1/CW	1C52- S6/CW	2C44-51	2C52-56#224	UR/TBP	PFcCN1	PFcCN2	TFcCN	1CFcCN	R', 52- S6	R', 59- S67	CWR, 50-61
Na		74421.3		84906	87763	91756	37520	85054	84503	108828	112478	59935	149106	27267	30241
Al		0		26886	18384	0	0	0	0	0	0	0	80416	56395	95185
Fe		0		8281.2	5053.3	14346	21538	0	85341	17208.4	16707.7	14386	17854	2323.8	13519
Cr		0		47.505	86.147	31.335	31.029	35.236	0	0	0	0	19.034	12451	86255
Bi		0		13892	8542.2	20861	30102	20690	0	43576.4	49573.1	0	25403	0	0
La		0		0	0	0	0	26692	0	0	0	0	0	0	0
Ce		0		0	0	0	0	0	0	0	0	0	0	0	0
ZrO(OH)2		0		673.46	506.03	0	0	0	0	0	0	0	1051.7	0	0
Pb		0		0	0	0	0	0	0	0	0	0	0	0	0
Ni		0		0	0	0	0	0	5296.88	3929.25	24844	4711.7	0	0	0
Sr		0		0	0	0	0	0	0	0	0	0	0	0	0
Mn		0		0	0	0	0	190.28	0	0	0	0	0	0	0
Cs		0		0	0	0	0	0	0	0	0	0	0	0	0
K		0		0	0	0	0	7789.5	0	0	0	0	0	0	0
balance															
density		1.48011		1.3952	1.2648	1.2672	1.1383	1.2323	1.5282	1.49782	1.52137	1.3884	1.5943	1.5334	1.9841
vol% solids		12		13.7	24.9	6.8	3.4	3.9	1.2	3.7	3.2	1.4	6.5	8.9	2.3
void frac.		0.5032		0.8026	0.7555	0.7506	0.9658	0.9251	0.5526	0.56193	0.49288	0.9942	0.2781	0.7056	0.6688
wt.% H2O		38.3466		65.494	73.61	70.904	81.073	68.962	30.449	32.8493	28.0527	81.588	57.788	56.029	38.078
TOC wt.%C		0		0	0	0	0	0.4152	0	0.64959	0.36973	1.8521	0.3474	0	0
free OH-		1413.58		28.312	73.834	193.87	218.81	468.48	60.802	152.367	67.4196	701.21	33.653	295.87	64.443
OH-		70327.2		59449	40478	13325	19938	468.46	99214	18341.8	21283.4	26242	17599	168648	220413
NO3-		2241.47		14160	19717	42965	42545	61618	65015	67454.7	58250.4	102120	5673.5	67381	60603
NO2-		0		3656.1	5090.7	0	0	0	0	0	0	5600.1	1431.2	0	0
CO3-		31987.6		0	0	0	0	0	4363.5	0	0	0	0	0	0
PO4-		30383.9		95307	68622	86076	26565	12477	4488.6	24511.2	26594.1	8841.4	164824	0	0
SO4-		56474.6		2632.7	3665.8	2778.5	2751.3	0	112329	116969	131968	1100.7	1336.1	680.86	848.28
SiO3-		40.6213		3048.1	2049.7	5981.9	4834.6	0	0	7981.57	9028.17	0	5231	0	0
F-		0		5507.9	4694	7386.1	2494.2	46554	0	5989.52	6428.09	0	7514.8	0	0
Cl-		0		0	0	0	0	0	296.26	0	0	0	0	0	0
C6H5O7-		0		0	0	0	0	0	0	0	0	0	0	0	0
EDTA-		0		0	0	0	0	0	0	0	0	0	0	0	0

	MW 44- 51	MW 52- 56	1C44- 51/CW	1C52- 56/CW	2C44-51	2C52-56	#224	UR/TBP	PFeCN1	PFeCN2	TFeCN	1CFeCN	R' 52- 56	R' 50- 57	CWR/AL 52-60
HEDTA---	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NTA---	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
glycolate-	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
acetate-	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
oxalate-	0	0	0	0	0	0	15227	0	0	0	0	0	0	0	0
TBP	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NPH	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
CCM	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
hexone	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NiFe(CN)6--	0	0	0	0	0	0	0	0	24415.6	13896.8	69613	13057	0	0	0
Pu (μCi/g)	0.00476	0	0.0045	0.1247	0.0092	0.0112	0.0061	0.0018	0.00086	0.00076	0	0.3179	0.0102	0.273	1.837
U (μg/g)	164261	758.9	1475.8	210.88	321.82	0	49947	16963	19212.1	47220	4468.2	2003.7	26660	25473	
Ce (μCi/g)	0.62888	0	5.6994	53.679	0.1038	0.2258	0	6.1105	7.1336	8.12054	1286.2	814.4	41.255	1761.9	1.3973
Sr (μCi/g)	52.661	0	0.3635	1.6656	0.3546	0.3188	0	186.65	29.4436	33.3497	0	4.2975	94.409	3466.5	14.467
									</						

TANK 111-T STATUS

<u>Liquid, Gal.</u>	<u>Sludge, Gal.</u>	<u>Sp. Gr. (g/cc)</u>
82 x 10 ³	430 x 10 ³	1.032

Chemical Analyses

Free NaOH	0.701N
CO ₃ ⁼ (g/l)	
AlO ₂ (g/l)	0.022
F ⁻ (g/l)	
Cl ⁻ (g/l)	15.8
Na ⁺ (g/l)	21.0
NO ₃ ⁻ (g/l)	2.47
pH	9.5
CN ⁻	N.D.

Gamma Scan

Zr-Nb ⁹⁵ (uc/l)	3.48 x 10 ²
Ru-Rh ¹⁰⁶ (uc/l)	1.21 x 10 ³
Sb-125 (uc/l)	N.D.
Cs-134 (uc/l)	N.D.
Cs-137 (uc/l)	1.15 x 10 ³

Boil-Down Studies

Percent Boil-Down	0	50	80	90
Boiling Point (°C)	105	105	106	107
Percent Solids -				
at boiling point	clr	clr	5	7
at room temperature	clr	clr	28	70

Atlantic Richfield Hanford Company



Date: June 7, 1974
 To: R. L. Walser
 From: R. E. Wheeler *R. E. Wheeler*
 Subject: ANALYSIS OF TANK FARM SAMPLES
 SAMPLE: T-3304 111-T

Vis-OTR: Clear, Yellow, 30% solids like rust. Filtrate <10 mrad/hr.

pH: 13.25

SpG: 1.018

OH: 0.254 M

Al: $< 6.48 \times 10^{-4}$ M

Na: 0.446

NO₂: 4.39×10^{-3} M

NO₃: 0.083 M

Pu: 1.17×10^{-6} g/l

DTA: No Exotherm

SO₄: 3.79×10^{-3} M

PO₄: 2.16×10^{-2} M

F: 3.56×10^{-2} M

CO₃: 0.024 M

GEA: ¹³⁴Cs - 1.13 μ Ci/gal
¹³⁷Cs - 3.69×10^{-2} μ Ci/gal
¹²⁵Sb - 4.66 μ Ci/gal

Water: 98.24%

Cooling Curve: 10°C for 45 min. No solids.
 5°C for 45 min. No solids.

30% Reduction - 27.5

Solids - 0

50% Reduction=50.0

Solids - 0.5%

REW:jd

Atlantic Offshore Hanford Company



Date: September 24, 1974

To: R. L. Walser

From: R. E. Wheeler *R. E. Wheeler*

Subject: ANALYSIS OF TANK FARM SAMPLES
SAMPLE: T-4893 111-T ←

Vis-OTR: Black. 90% solids. 10 mrad/hr, filtrate

pH: 12.9

SpG: 1.0202

OH: 0.206 M

Al: 7.75×10^{-4} M

Na: 0.188 M *new*

NO₂: 5.17×10^{-3} M

NO₃: 0.109 M

Pu: $< 1.41 \times 10^{-6}$ g/l

DTA: No exotherm below 200°C

SO₄: 4.43×10^{-3} M

PO₄: 2.33×10^{-2} M

F: 4.28×10^{-2} M

CO₃: 6.59×10^{-3} M

GEA: ¹³⁷Cs - 5.72×10^2 μ Ci/gal

Water: 95.45%

Stilling Curve: 40°C for 45 min. 90% Solids.
35°C for 45 min. 90% solids.
30°C for 45 min. 90% solids.
25°C for 45 min. 90% solids.
20°C for 45 min. 90% solids.
15°C for 45 min. 90% solids.
10°C for 45 min. 90% solids.
5°C for 45 min. 90% solids.

REW:jd

cc: MR Christensen
CS Buckingham

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